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Calamine lotion;
its place in cosmetics and medicine

Chemical structure and
antimicrobial activity of Bis-phenols

Oligodynamic silver and
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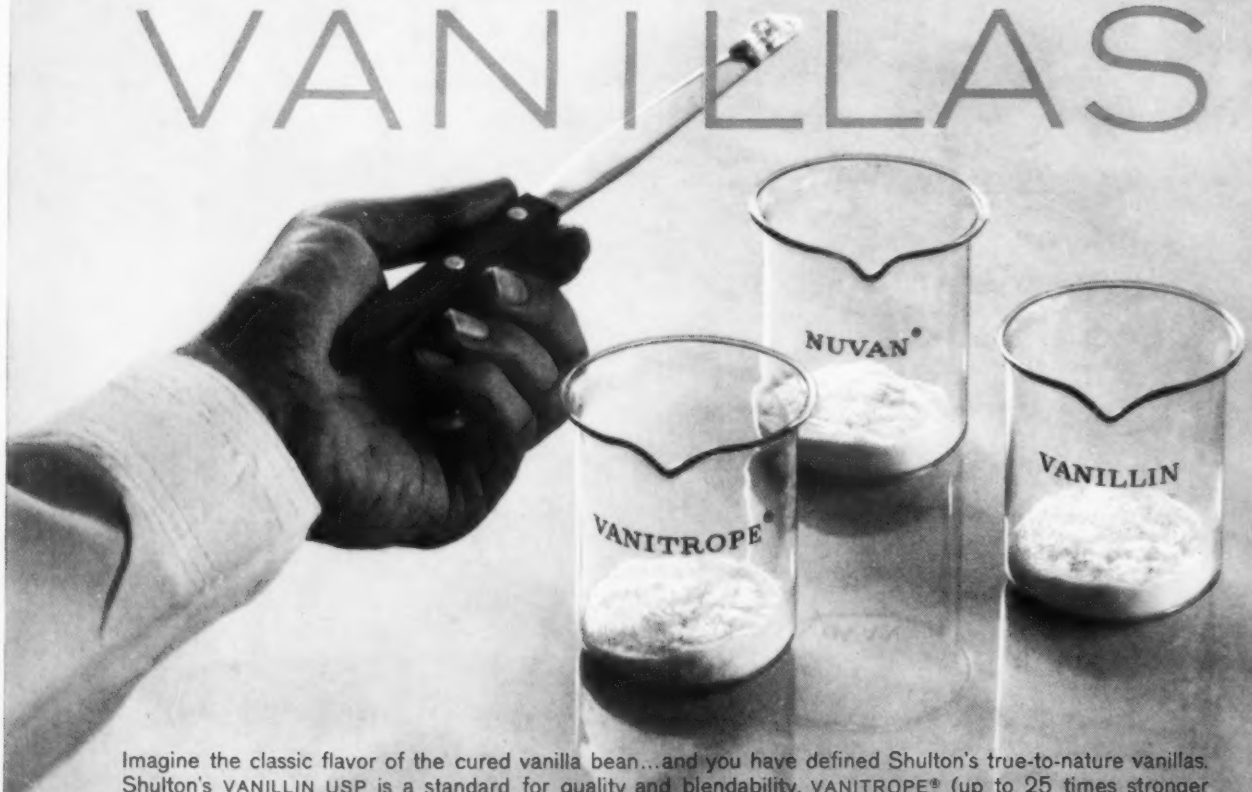

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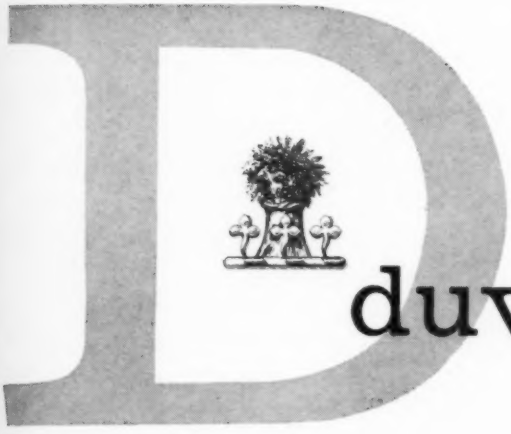

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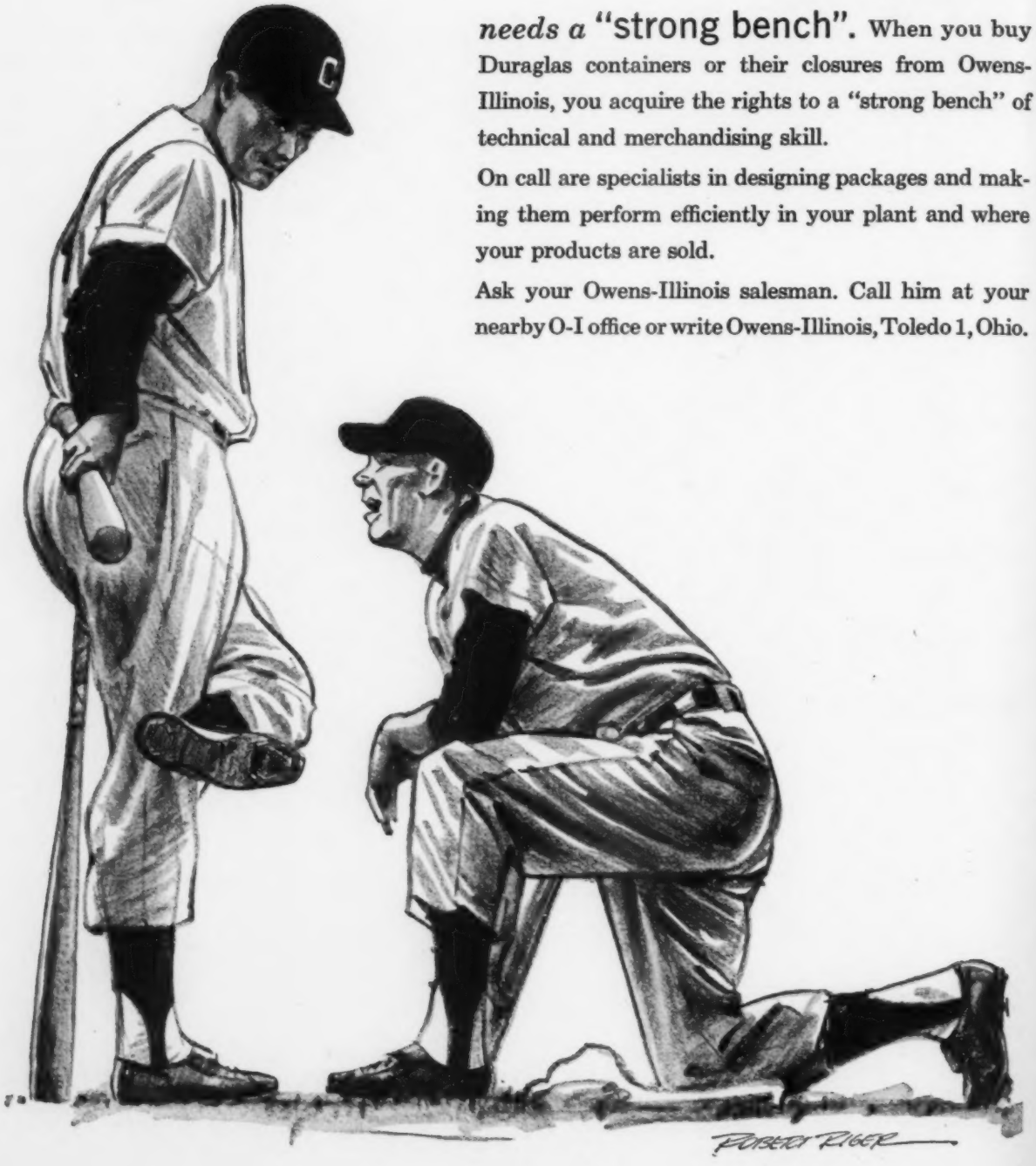

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American Perfumer

VOL. 75, NO. 9 SEPTEMBER, 1960

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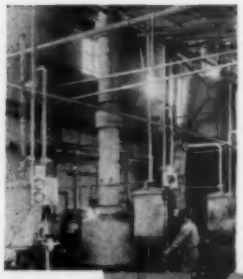

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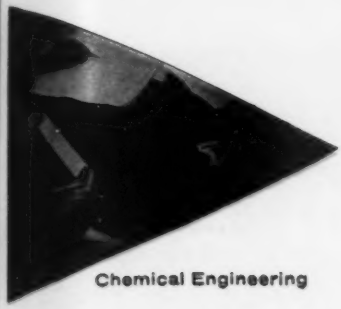

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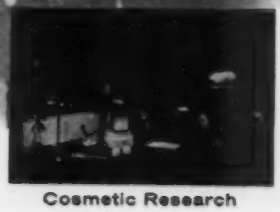

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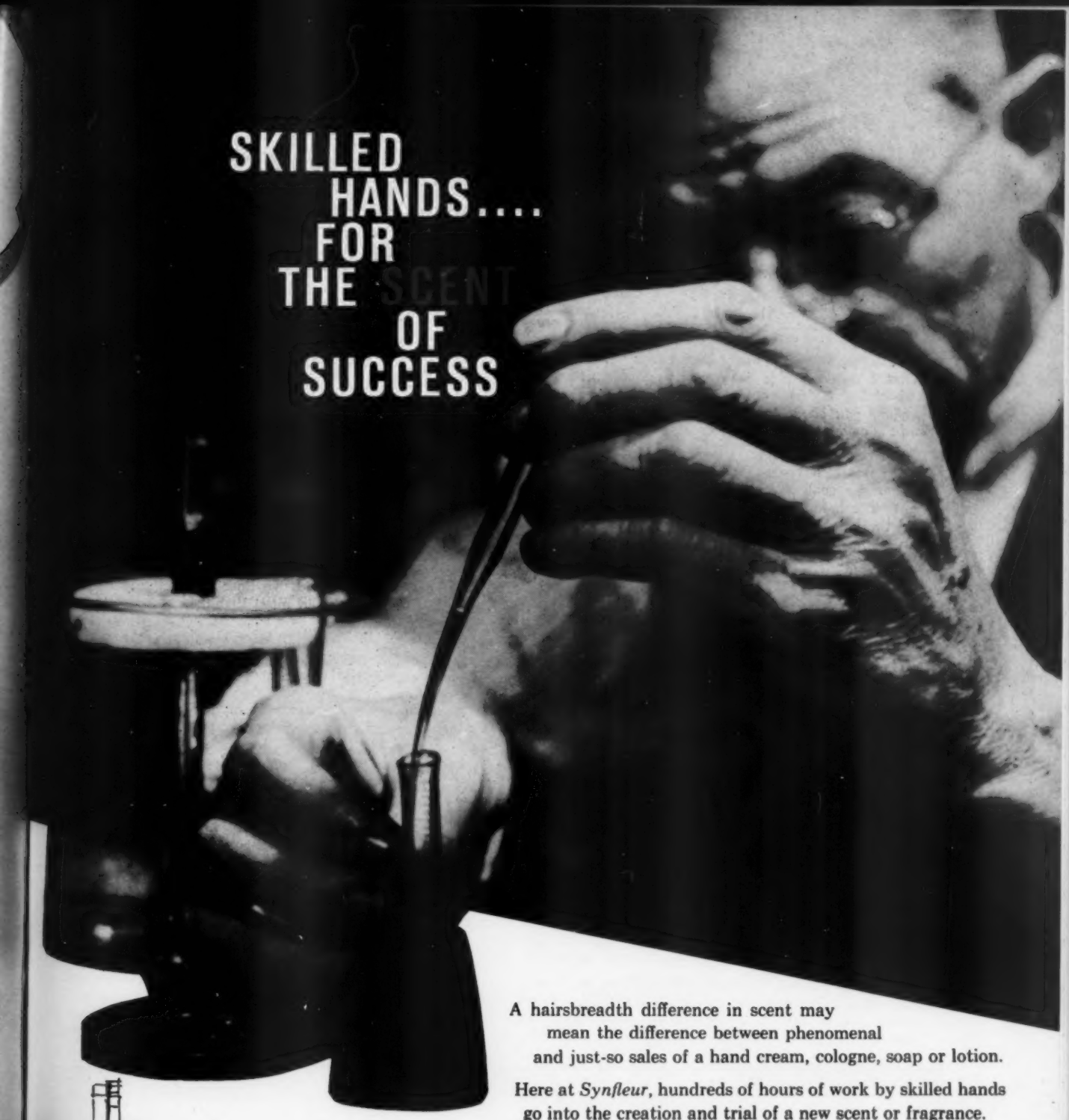

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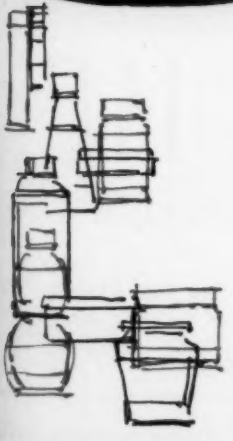

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Questions and Answers

Q. *I do not know whether you have available or are free to dispense the information I request, but if so I would appreciate your consideration very much. What I would like is a basic formula for restoring natural looking color to gray hair. If not a formula, perhaps you could tell me the coloring agents used and the amount. H. E. A., Wisconsin.*

A. The stock formula that we can give you for the products which are referred to as "color restorers," is one per cent of the lead acetate, two to 5 per cent of colloidal sulphur, five per cent of glycerol and the balance of water and perfume. This formula can be modified in various ways to suit your needs. We suggest that you go to the main public library and get one of the several books on cosmetics which they have there for further suggestions. These products do *not restore the color of hair.* Nothing but your own body can restore the color of hair. These products and all products labelled "color restorers," are hair dyes.

Q. *In a recent issue you had an article on cold wave fixatives. We are very much interested in this fast action of neutralizing. We would like more information on this formula, where can it be purchased or if you can give us additional details. V. S., Colorado*

A. We do not know who makes the cold wave fixatives described in "Desiderata" in the U.S.A. They are both patented in Austria. We regret we cannot help you further.

Q. *We would appreciate knowing the name and address of a company from whom we could buy small quantities of the following ingredients for use in face creams: spermaceti, Protagin X, cetyl alcohol, and Tegocept P. Also can you tell us the reason for the oils in a face cream to bleed to the top of the cream after it has been made and stands for a few days? C. V., Michigan*

A. We have given you the names of suppliers by letter. The separation of an oil on the surface of a face cream can be due to a number of causes such as poor formulation, insufficient emulsifier or stabilizer and faulty manufacturing methods. Storage of finished product in excessively hot or cold warehouses may cause some oil separation too.

Q. *We are interested in preparing a cream to be sold on the market in retail sources. Will you be kind enough to let me know if it is permissable to use the words "Wrinkle Remover" on the face of the label? I am also interested in a good formulation for a liquid eye liner and a liquid moisturizing lotion. K. C., Pennsylvania*

A. You may not use the words "Wrinkle Remover" on the labelling of your products. There is no such thing. It is impossible to remove wrinkles. You may soften the lines of wrinkles, but you cannot remove them except by surgery, if then. In regard to the formulation of a liquid eye liner and a liquid moisturizing lotion, keep in mind that there are so many different kinds that it is difficult to determine exactly what you have in mind. As to the liquid eye liner, it would be our guess that what you would want is a mixture of the pigments to give the necessary shade and then suspend these in water with sufficient amount of bentonite or similar suspending agent, or perhaps one of the water soluble gums, such as methyl cellulose or an ethoxylated cellulose. We suggest you try the following formula for the liquid moisturizing lotion:

Liquid Lanolin absorption base	14%
Light mineral oil	30%
Sorbitol syrup	3%
Glycerol	2%
Borax	0.3%
Water q. s.	100%

The fatty materials are melted together and brought to 70°C. The water soluble materials and borax are heated to the same temperature. Add the water to the fats under constant agitation. You can increase the moisturizing capacity by increasing the amounts of both glycerol and sorbitol, possibly adding some propylene glycol if you wish. You could also use one of the polyethylene glycols of molecular weight 400.

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Notes

Bob (R. P.) Scherer who with brothers and dad developed and manufactured soft gelatin capsules by a rotary process, died at 53 years of age. . . . . Speaking of capsules, F.D.A. came through with a 1.0 mg. tolerance for Cobalt in dietary supplements. . . . . Looks like quite a pat on the back for P & G Crest tooth paste from the American Dental Association. But how about the Crest TV commercial that opens with a child shouting to its mother, "Look, Mom, no cavities" and the mother answers, "No *new* cavities?" Or am I too fussy about this play on words? . . . . . Beef may be tenderized on the hoof according to a news item in C & EN for May 16. Just inject the animal with a special papain extract . . . . British Patent No. 837,521 describes a method of removing hair from skins by means of ultrasonic waves of a frequency yower than 100 kc/s . . . . . Rhone-Poulenc has patented gamma-cyclohexylcrotonaldehyde as a perfume "enhancing" compound . . . . . Hoffmann La Roche have a patent on such compounds as cis-1-acetoxy-3, 7-dimethyloctene as fruity or rose-like aromatics . . . . and while on aromatics it is a compliment all the way around to Polak & Schwarz International N. V. and Firmenich to see them agree on the pooling of Patent rights in the manufacture and sale of a new indane musk . . . . . In answer to a question, the J.A.M.A. Editors state that when the oral Lactobacillus count is in excess of 10,000 per cc there is high caries activity . . . . .

I.F.S.C.C. MUNICH CONGRESS

Under the sponsorship of the International Federation of Societies of Cosmetic Chemists, the German Society of Cosmetic Chemists is holding an International Congress at Munich on August 31 through September 3. About two dozen Yankees are attending to hear twenty scientific papers on subjects ranging from psychology analysis, application, ingredient activity through a study of hair, its waving and dyeing.

Other Scientific meetings will be held during the stay of the U.S. members at London, Geneva and Barcelona.

The S.C.C. in the United States arranges these tours for every second year to enable U.S. members to hear a great many scientific papers on one jaunt.

Plant tours and entertainment complete the activities.

Such an intimate contact with cosmetic scientists of all European countries, enables U.S. delegates to learn at first hand the progress made abroad. Subjects considered questionable here are actively explored by everyone concerned.

This meeting opens a new era in Cosmetic Science. Those who are unable to attend the first International Congress may be able to do so in two years.

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Does romance have a fragrance? Of course it has — and for each of us apart. It can smell sweet as a persimmon, soft as down on cheek, warm as May wine, tender as a kiss. How much a part of happiness — and all our moods — are the scents of things we love!

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products are based on dihydroxyacetone (DHA) as the active ingredient.

At least one patent is pending and possibly others. Users should be aware of this. A German Patent #669,157 (1938) mentions dihydroxyacetone as a *tanning* agent for tablets coated with gelatin.

Barmak authored an article for the AMERICAN PERFUMER last March (p 57) in which he delved into the origins, chemistry, manufacture, and uses of dihydroxyacetone. The editors suggested an alcoholic lotion containing 2½% of the material as an effective "instant tan".

Since that time, several publications have appeared, evaluating the ingredient, its properties and uses. Bryce (*Pharm. J., 184,* 491, (1960)) reviews the subject and lists a number of references. Mumford (*Brit. J. Dermatol., 72,* 279 (1960)) evaluates a lotion made from the following:

Dihydroxyacetone	2.5
Glycerin	2.5
Methylated Spirits (denatured alcohol)	33
Water to make	100

Six to ten hours after application, a light brown "pigmentation" is produced. Hourly applications may result in a "light negroid tint". The color produced disappears in five to eight days.

After applying the above formulated lotion to the skin of 32 normal and 10 eczematous skins, no discomfort or irritation was produced. Horny skin showed deeper and longer lasting browning. The "depth" of tint varied considerably.

The skin treated with the DHA tanning solution was just as susceptible to solar sunburn as untreated skin, although the erythema was not as noticeable.

Vitiligenous areas responded to the coloring solution as well as normal skin areas. Matching skin shade was problematical, however.

Mumford concludes by noting that it is difficult to get an even tan coloration over wide areas. He anticipates possible severe reactions resulting from over-exposure.

The difficulty of getting an even tan has been mentioned by others described by this writer previously.

As this is being written, Kanas and coworkers are giving two papers at the A. Ph. A. meeting in Washington August 18. Reactions of DHA with skin and certain amino acids, proteins, metaproteins, globulins, prolamines, albumins, protamines, and simpler proteins were carried out. The nature of the tan coloration reaction has been studied.

Vehicles were also studied for their influence on the tanning effect of DHA on skin. Such factors as solvents, pH, surfactants, w/o and o/w emulsions were among the factors considered. Results were recorded photographically.

Dihydroxyacetone (DHA) is readily available from at least three sources at prices as low as around $6.00 per pound in contract quantity. More will be heard about it in dermatological journals without doubt. Conventional sun tan preparations containing sunscreens are now also fortified with DHA to get a "tan" and yet protection from sunburn.

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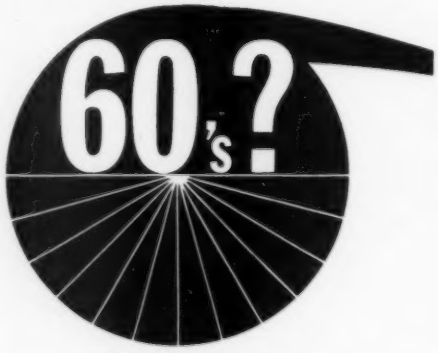

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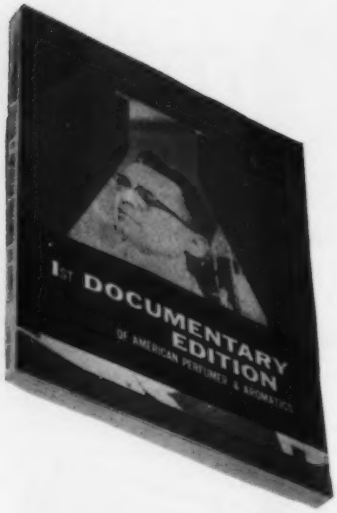

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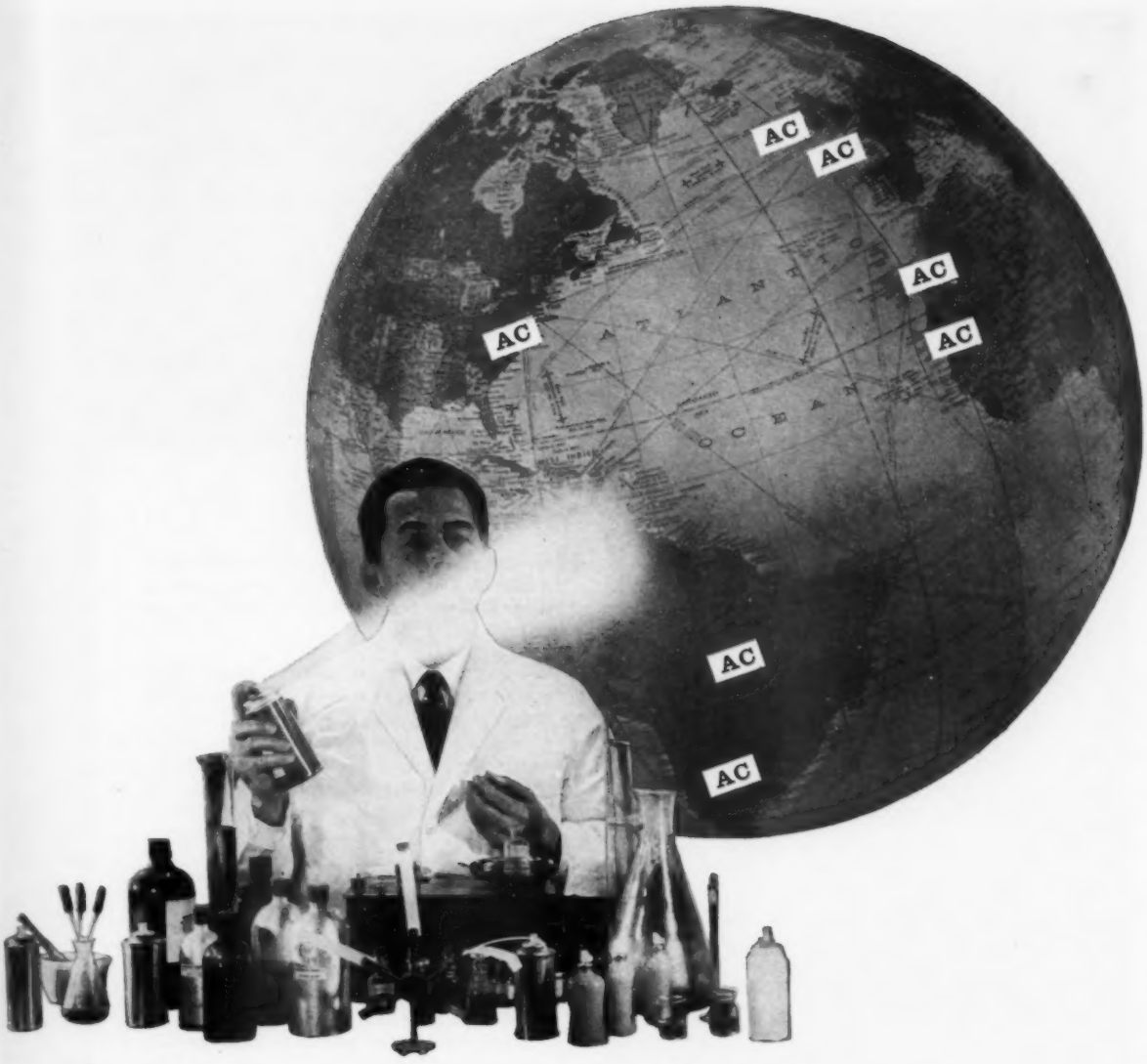

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Calamine lotion;

its place in cosmetics and medicine

BY H. GEORGE DEKAY
Professor of Pharmacy
Purdue University, School of Pharmacy

Calamine lotion has been known for over two thousand years. It has been studied and reported in the literature for the past two hundred years. It was discussed in the British literature both pharmaceutical and medical in the early part of the last century. The literature covered the adulteration and substitution in preparations used in the treatment of skin infections. The controversy led to a general decision that it would be better to use an artificial calamine of definite chemical composition for the natural product whose composition varied with each batch of material.

Calamine has been defined as native zinc carbonate. The synthetic product, "Prepared Calamine", was first introduced into the National Formulary Fifth Edition (1926) and defined as "zinc oxide with a small amount of ferric oxide, and contains, after ignition, not less than 98% of zinc oxide". It remained official in the National Formulary until 1946 when it was introduced in the U.S.P. XIII with the same definition as used in the National Formulary.

Depositis of Native American Calamine were exhausted about fifty years ago; authentic samples are scarce and hard to get at the present time. China still has a great abundance of Native Calamine, but shipping costs would make it exorbitant to use.

Creech and Lee (1) compiled a comprehensive thesis on natural and artificial calamine. They considered its source and composition from a chemical, therapeutic and pharmaceutical standpoint. According to the *Encyclopedia Britannica*, "Calamine was used indiscriminately for the carbonate and the hydrous silicate." The minerals closely resemble each other in appearance and can usually be distinguished only by chemical tests. Calamine was named by James Smithson in 1803 (2). In 1832, F. S. Beudant (2) restricted the name to the hydrous silicate and Smithsonite was used for the carbonate. Edward Dana, a minerologist of that period, adopted these meanings for the two ores. However, in England (following Henry Brooke and William Miller 1852) those designations have been reversed and calamine was used for the carbonate.

There is a difference in English and American nomenclature, as zinc carbonate is still called calamine or zinc spar by the English and Smithsonite by Americans. The zinc carbonate was the one commonly accepted in medicine in the early days. The New London Dispensary (1716) discussed the therapeutic properties of calamine. It was reported to dry, bind, fill ulcers with flesh and, sprinkled upon galled places in children, to dry and heal them.

According to Strother (1727) Lapis calaminaris was cooling and drying when the powder was sprinkled on wounds and ulcers. Turner (1667-1740), a London surgeon, invented calamine cerate (Turner's cerate). His preparation was of good consistency and a true cerate. It was used as a plaster.

According to Creech and Lee (3) they arbitrarily set the date of 1800 as the beginning of the modern period in the history of calamine. The history of calamine since that time is both interesting and enlightening, and one can assume that this product has a definite place in our dermatological medicine because of its long and extended use.

During the period of 1800 to 1866 the samples of calamine seemed to be composed of barium sulfate with some oxide of iron and other impurities followed by combinations of zinc oxide with barytic calamine. In 1874, Rosenstadt (4) advised the use of an artificial calamine consisting of one part of red oxide of iron and twenty parts of zinc carbonate to replace the

product then in use. There were a few who championed Native Calamine in place of either zinc oxide or zinc carbonate artifically colored.

Barrie (5) in 1900 discussed the calamine problem and stated that many samples in use would not completely dissolve in dilute acids and that the majority contained barium sulfate or calcium sulfate in addition to the iron oxide for coloring the samples in use. Wild (6) stated that lotions which approached the normal skin color had a valuable place in dermatological medicine.

When one examines the official compendium, we find that calamine was mentioned in the first United States Pharmacopoeia and continued in and out of the U.S.P. in some form since that time. The first National Formulary of 1888 contained a formula for calamine ointment which contained prepared calamine. The National Formulary II and III contained the calamine ointment using prepared calamine. However, the National Formulary IV contained a monograph on prepared calamine and defined it as "native zinc carbonate containing a varying amount of zinc silicate, calcined at a moderate temperature, or calcined zinc carbonate containing a small amount of ferric oxide".

The next National Formulary contained a monograph on Prepared Calamine and defined it as "zinc oxide containing a small amount of ferric oxide and silica. It contains after ignition not less than 98% zinc oxide." It was at this time that a lotion of calamine was introduced, along with calamine liniment and a compound calamine lotion. The Sixth Edition of the National Formulary had few changes in the monographs. The compound calamine lotion was now changed to Phenolated Calamine Lotion. Prepared calamine first appeared in the British Pharmaceutical Codex of 1907 and remained as such until 1934 when it defined prepared calamine as basic zinc carbonate with or without the addition of zinc oxide, suitably colored with ferric oxide. At this time they included calamine liniment, compound calamine liniment, calamine lotion, and calamine ointment.

Calamine in the form of the lotion has been used to a much greater extent since its introduction than the calamine ointment or liniment. There have been many changes in this preparation since its introduction in the National Formulary. The problems studied have been of the stability of the preparation as well as the use of phenol in phenolated calamine lotion. It has been the subject of many papers since its adoption.

It is well to study just why this preparation has had such long and continued usage. It seems that calamine as such was used by the ancients in all forms of skin ointments. It has persisted into the present era. The dermatologist of today is using calamine or calamine lotion for a number of skin ailments. Among these are poison ivy, as an aid in drying up the blisters and to soothe the skin, poison oak, skin itching, sunburn, insect bites, as well as a number of other conditions. They have also incorporated calamine in ointments to give the preparation a skin color.

Meyer (7) studied the action of calamine lotion on the bacterial flora of the skin. The skin on the upper and lower surfaces of the arms was used for the experiment. The area was first washed with soap and water, then dried, after which a sterile cotton was moistened in sterile saline and rubbed on one inch of the arm. The swab was then rubbed over an agar plate for control. The lotion was now applied with another sterile swab to each of 4 remaining square inch areas. After 30 minutes, the lotion on the second square inch was gently washed off with a sterile swab and discarded. The area was now swabbed and the swab rubbed on an agar plate. This was repeated on the remaining three squares after 1, 1-1/2 and 2 hours, respectively. Cultures were taken at these variables of time and the plates incubated for forty-eight hours and counts were made using a Frost plate counter. After the 1/2 hour treatment with calamine lotion, the bacterial count was reduced as much as 75 to 85%.

Three different calamine lotions were used in this study as follows: (1) zinc carbonate lotion, (2) prepared zinc oxide lotion, and (3) Bentonite Cuticolor Lotion. All three were active in markedly reducing the bacterial flora.

The Editor of the *Pharmaceutical Journal* (8) in discussing the use of calamine lotion as a sunburn lotion, states that "it may be classified as a liquid face powder containing more zinc oxide. It is astringent, removing roughness and redness due to exposure and the action of excessive sunlight". It is also assumed to be a means of combating many of the various types of itching and burning sensations experienced during the summer months. This would naturally include many of the ailments listed above. One manufacturer whose preparation contains an antihistamine has recommended it for rashes such as diaper rash, cosmetic rash, and prickly heat rash.

Bliss (9) mentions the use of "Pink Lotion" which is calamine lotion, for the relief of severe itching. Dyniewicz and Fantus (10) found means of overcoming faults in calamine lotion and presented twelve new or improved calamine preparations using the name "Cuticolor for Calamine". They found that calamine lotion was a very good protective against the actinic or ultraviolet rays. They also brought out that a variety of skin diseases and irritations were improved or cured by calamine lotion. These coworkers prepared two lotions as follows:

A		*B*	
N.F. VI Calamine Lotion		Improved Calamine Lotion	
Prepared calamine	80 Gm.	Calamine	140 Gm.
Zinc oxide	80 Gm.	Bentonite	30 Gm.
Glycerin	20 cc.	Zinc sulfocarbolate	40 Gm.
Calcium hydroxide solution to make	1000 cc.	Glycerin	40 cc.
		Witch Hazel Ext.	200 cc.
		Water	550 cc.

These lotions were used in a clinical study with the following results:

Ailment	A	B
Dermatitis		+
Intertrigo	+	+
Infectious dermatitis		+
Prickly heat	+	+
Poison Ivy	+	+

External irritations	+	+
Acne		+
Urticaria	+	+

+ = improved

In many instances the patient did not return after treatment, which should indicate an improvement.

Hilton (11) stated that calamine lotion is used largely by women on their face, neck, hands and arms in ivy poisoning. The slight pink color is more like the cosmetics they use and would show opposition to the brown color of calamine. This color was obtained by mixing zinc oxide with a mixture of red ferric oxide and yellow ferric oxide usually giving a brown color.

Fantus and Dyniewicz (12) suggested a new calamine known as Cuticolor stating that "Cuticolor preparations should in general be preferred in dermatology to those with other colors, and that the use of skin colored preparations is mandatory, so far as possible, on surfaces of the body exposed to view." They presented a formula for a cuticolor powder as follows:

Red ferric oxide	3.0 Gm.
Yellow ferric oxide	4.0 Gm.
Zinc oxide	93.0 Gm.

They stated that by incorporating cuticolor powder in a lotion it could be made to more nearly approach the color of any skin. King and Becker (13) in their review of calamine brought out that the first formula for calamine lotion appeared in the University College Hospital Pharmacopoeia published in London in 1873. In studying earlier reports they found that in 1848 it had been suggested to substitute either pure zinc oxide or zinc carbonate suitably tinted with iron for the natural calamine.

The 1905 Compendium of the Canadian Formulary included a formula for calamine lotion made with water or rose water as the vehicle. This same formula was repeated in the Canadian Formulary of Unofficial Preparations in 1908 and 1910.

The first official calamine lotion appeared in the National Formulary V. This formula was:

Calamine	8 Gm.
Zinc oxide	8 Gm.
Glycerin	2 cc.
Lime water qs	100 cc.

This was left unchanged in National Formulary VI, but was revised in the National Formulary VII as follows:

Calamine	80 Gm.
Zinc oxide	80 Gm.
Glycerin	20 cc.
Bentonite magma	400 cc.
Lime water qs	1000 cc.

Sage reported that glycerin would prevent the powders from blowing off the skin when the lotion was applied and lime water was included to neutralize acid perspiration.

The United States Pharmacopoeia XIII retained the formula with bentonite magma, but the U.S.P. XIV revised the formula to the following:

Calamine	80 Gm.
Zinc oxide	90 Gm.
Polyethylene Glycol 400	80 cc.
Polyethylene Glycol 400 Monostearate	20 Gm.
Water	900 cc.

This formula was again changed back to bentonite magma in U.S.P. XV. The revised formula is as follows:

Calamine	80 Gm.
Zinc oxide	80 Gm.
Glycerin	20 ml.
Bentonite Magma	250 ml.
Calcium hydroxide solution, a quantity sufficient to make	1000 ml.

This is the present official formula for calamine lotion and will be the one retained for U.S.P. XVI.

Several variations for calamine lotion have been recommended for specific and general external use. One formula which appeared in the *Pharmaceutical Journal* recommended that the lotion could be prepared extemporaneously. This formula was: (13)

Zinc carbonate, ppt.	10.0 Gm.
Zinc oxide, BP	5.0 Gm.
Dark ochre	0.2 Gm.
Golden ochre, extra strong	0.1 Gm.
Glycerin	5.0 cc.
Phenylethyl alcohol	4.0 gtts.
Patchouli oil	1.0 gtts.
Rose water, undiluted	30.0 cc.
Distilled extract of witch hazel	30.0 cc.
Distilled water qs	100.0 cc.

Bliss (9) used the following formula to allay severe itching:

Phenol	1.6 Gm.
Prepared calamine	8.0 Gm.
Zinc oxide	8.0 Gm.
Glycerin	2.0 cc.
Lime water qs	100.0 cc.

He advised that the film left on the skin by the lotion should be removed with a fixed oil.

The symposium on calamine which was sponsored by the Bulletin of the National Formulary Committee recommended the following modified formula for phenolated calamine lotion:

Phenol	dr xx
Calamine	dr i
Zinc oxide	dr ii
Glycerin	dr iii
Aqua Rosa qs	oz iv

Fantus and Dyniewicz (12) recommended a formula using 3% bentonite with witch hazel extract in this same symposium. At that time they recommended coloring zinc oxide making their "Cuticolor" powder.

Rosen (14) prepared a series of calamine lotions with 2% bentonite and varied the pH by using different amounts of lime water. The amount of sedimentation was determined by measuring the supernatant liquid and the pH with a Beckman pH meter. The lotion that showed the least amount of separation

was obtained by using 5 cc. of lime water and 90 cc. of distilled water and had a pH of 9.00.

Broughton (15) substituted 25% acetone for lime water in the official formula in an attempt to have the lotion dry more quickly after application. He also prepared phenolated calamine lotion in the same way. Another lotion was prepared with and without glycerin. No difference could be ascertained between the two lotions.

Goodman (16) favored the formula with magma bentonite. He did not like the "atrocious" pink color of calamine but favored the more neutral color of neocalamine. He recommended that the lime water be left out of the formula using distilled water in its place. The following formula was suggested:

Stearic acid	4 Gm.
Triethanolamine	1 Gm.
Distilled water qs	100 cc.

To this formula could be added various combinations of powder such as: prepared calamine and zinc oxide 8 Gms. each; or, prepared neocalamine 15 Gms.; or prepared neocalamine 8 Gms. and zinc oxide 4 Gms. He also stated that the powders would settle rather slowly from the completed lotion.

The Editor of the Practical Edition of the *Journal of the American Pharmaceutical Association* (17) suggested that a stearate soap of triethanolamine and stearic acid, the same as the Goodman formula, be used as a vehicle for calamine lotion. He did not like the formula which contained bentonite magma because it stuck to the sides of the bottle.

The Editor of *Drug and Cosmetic Industry* (18) in replying to a reader's question concerning calamine lotion, stated that zinc oxide would react with a soap emulsifying agent and destroy the emulsion. The following formula was recommended to keep the powders suspended:

Cetyl alcohol	1.0 part
Sodium lauryl sulfate	0.5 part
Glycerin	5.0 parts
Water qs	100.0 parts

It was suggested to grind the calamine powder in the presence of this vehicle and that additional stability may be obtained by adding 0.5% of a gum.

Ohmart and Stoklosa (19) prepared a series of calamine lotions by increasing the bentonite magma by 50 cc. increments from 0 to 500 cc. The relative viscosity was determined by noting the time required for the lotion to flow from a 50 cc. pipette. The rate of sedimentation was determined by allowing the lotions to stand in 100 cc. graduates. Sedimentation amounts were checked at various intervals of 15 minutes, 45 minutes, 2 hours, twenty hours, one and two weeks. The rate of the settling and the accuracy of dispensing were related to the amount of bentonite magma used.

Nadkarni and Zoph (20) studied the various suspending agents that could be used for making calamine lotion. They experimented with the following suspending agents: cellosize, pectin, sodium alginate, and polyethylene glycol 400 monostearate. The agent giving the most satisfactory lotion was polyethylene 400 monostearate in a 2% concentration using lime water as the vehicle. The particle size of the powders, viscosity and method of preparation were some of the factors that were found to affect the rate of settling. The lotion was also found to possess a lower order of irritation and sensitizing ability.

Another emulsified vehicle suggested by Dean, Brodie and Chin (21) was as follows:

Cetyl alcohol	36 Gm.
Sodium lauryl sulfate	4 Gm.
Distilled water qs	1000 cc.

This emulsion had suitable keeping qualities and was used to suspend calamine, titanium dioxide and white lotion, N.F.

Huyck (22) used "Veegum" to suspend the powder in calamine lotions and chalk mixture. A calamine lotion made with 5% Veegum had a pH of 10.6 and separated 12% in one week. The viscosity was determined by comparing the time required for the lotion to run out of a 50 cc. pipette as related to distilled water.

Marcus and DeKay (23) prepared calamine lotion using methyl cellulose and sodium alginate as suspending agents. The following formula was used:

Zinc oxide	80 Gm.
Calamine	80 Gm.
Glycerin	20 cc.
Mucilage qs.	1000 cc.

The mucilages studied were 2% sodium alginate, 3% methyl cellulose, 1.5 cps., 4% methyl cellulose 15 cps., 1% methyl cellulose 100 cps., 2% methyl celullose 100 cps. and 1% methyl cellulose 400 cps. The particle size was measured with a calibrated microscope and viscosity was measured with a Brookfield Viscosimeter. They concluded that sodium alginate 2% was the best suspending agent of those used in this study.

Stanko and DeKay (24) studied the phenolated calamine lotion in an attempt to determine the basis for the incompatibility to phenol and to modify the formula in such a way as to overcome this incompatibility. They studied the rate of sedimentation by placing 100 cc. of the lotion in a 100 cc. cylindrical graduate and noting the rate of settling using as their control the U.S.P. XIV and N. F. IX lotions. The particle size was measured by a microscope with a calibrated scale using a magnification of 430 diameters. The results of the above showed that the plain lotion had made no separation after two weeks but was so thick it would not pour. The phenolated lotion decreased very rapidly in viscosity as there was a separation of 12 cc. after four hours. Five days later this solution had separated into several powder layers and a yellow oily globule floated on the surface. The particle size was measured after 24 hours with little or no change in the plain lotion, but the phenolated lotion had particles ranging from 35-100 microns.

The pH was measured with a Beckman pH meter and all samples were thoroughly mixed before the pH readings were taken. The pH of the plain and phenolated lotions remained at pH 7 after two weeks.

Viscosity measurements were made with the Brookfield Viscosimeter and the necessary spindle number was rotated at 20 rpm at a temperature of 25° C.

The viscosity of the plain lotion taken immediately after preparation was 1750-1000 cps. The viscosity seemed to rise after 15 days to 2400-2500 cps. The viscosity of the phenolated lotion was 10-14 cps and remained at that viscosity.

After considerable investigation it was found that the following formula was satisfactory:

Calamine	320 Gms.
Zinc oxide	320 Gms.
Glycerin	80 cc.
Tween 80	20 cc.
Veegum 5% magma	1000 cc.
Sodium alginate 2%	200 cc.
Distilled water qs	4000 cc.

The following medicaments were incorporated in this new formula: 2% resorcinol, 1% liquefied phenol, 1% thymol, 1% thymol and 0.5% menthol, 1% camphor and 0.5% menthol, and 5% benzocaine. None of these lotions showed any signs of incompatibility or caking after 10 days. The lotion had a pH of 8.6 and a viscosity of 840-880 cps when measured on a Brookfield Viscosimeter. Spindle #2 was used and rotated at 20 rpm with a temperature of 25° C. Your author had an interesting experience at the U.S.P. Headquarters in New York City two years ago when this lotion which had been standing on a shelf at the Headquarters for three years or more was shaken once with a complete mixing and no caking or sedimentation showed when the bottle was inverted. This lotion has been used in the School of Pharmacy Apothecary for dispensing to the students of the University since 1952 with highly satisfactory results.

King and Becker (13) compared calamine lotion using different types of equipment such as Eppenbach colloid mill, Manton-Gaulin homogenizer, Lightnin mixer, mortar and pestle, and a Readco vibrating sifter. Their formula was as follows:

Calamine	40 Gm.
Zinc oxide	40 Gm.
Glycerin	10 cc.
Magma or Mucilage	200 cc.
Distilled water qs	500 cc.

Only U.S.P. Calamine and zinc oxide were used for this work. Those lotions made by hand were passed through a hand homogenizer and those made with the colloid mills were mixed with an electric mixer prior to passage through the mills. They found that all of the lotions made with a 100-200 mesh powder were gritty and unsatisfactory, while those made with the 200-300 mesh produced satisfactory lotions, but with less stability. It was found that the lotion prepared with the Manton-Gaulin produced the most stable product and that hand homogenizing the lotion prepared with the mortar and pestle improved the stability. They also found that separation increased at increased temperatures.

Goldstein (25) working at the A.Ph.A. Headquarters Laboratory, because of many complaints about the U.S.P. XIII formula made with bentonite magma, found that a new formula produced what seems to be the answer to the problem. The formula is as follows:

Calamine	80
Zinc oxide	80
CMC-M	20
Dioctyl Sod. Sulfosuccinate	0.65
Glycerin	30
Water	950
To make	1000 cc.

This lotion did not foam and poured easily, spread smoothly, and dried to form a thin film that did not rub off.

Etchells (26) reported a new formula for calamine lotion in 1954. He found that a calamine lotion containing a wetting agent and carboxymethyl cellulose as a suspending agent had advantages over the present and past lotions. The experimental lotion was of a thin cream-like consistency and was easily redispersed after storage on shaking. It spread easily, dried fairly rapid, and left a thin film. His formula was as follows:

Calamine	15
Zinc oxide	5
Sod. carboxymethyl cellulose	1
Diisopropyl napthalene sulfonate	0.05
Glycerin	5
Liq. Phenol	0.5
Dist. water qs	100.00

Mathies and Guth (27) reported that the official calamine lotion produced hydrogen peroxide in the package upon standing. They further showed that by irradiating the lotions with an ultraviolet light hydrogen peroxide could be produced. They purchased 10 brands of calamine lotion from the open market and irradiated each lot after which they classified these 10 brands into three categories, namely (1) 3 lots produced 0.461 mg. of H_2O_2 for each 5 Gm. of lotion, (2) a second group produced .114 mg. of H_2O_2 per 5 Gm. of lotion, (3) a third group produced .031 mg. of hydrogen peroxide, while the U.S.P. XIV formula produced .432 mg. of hydrogen peroxide in 5 Gm. of lotion.

Reese and Guth (28) showed that the presence of this hydrogen peroxide enhanced the bacteriostatic activity.

Minardi and Guth (29) worked with three formulas of calamine lotion: (1) N.F. VI, (2) U.S.P. XIII, and (3) U.S.P. XIV. Each of the three samples was irradiated with ultraviolet light and assayed for hydrogen peroxide according to the method employed by Reese. These were assayed after 3 days, then every two weeks for eight weeks. The results found were maximum production of hydrogen peroxide as follows: N.F. VI after 4 weeks with slight decrease thereafter, while in the phenolated lotion the maximum production was after three days with a gradual decrease at each period. The U.S.P. XIII lotion reached a maximum production after 3 days, holding constant to two weeks with no production thereafter, while the phenolated calamine showed a maximum production after three days with steadily decreased quantities thereafter. The U.S.P. XIV formula reached a maximum after two weeks, while the phenolated lotion required 4 weeks to reach a maximum production.

The proposed formula for U.S.P. XV was less effective in producing H_2O_2 when irradiated than the N.F. VI formula. They found that both Sodium CMC and Dioctyl Sodium Sulfosuccinate depressed this property to form H_2O_2.

Escabi and DeKay (30) worked with calamine lotion in 1956 to investigate the influence of a number of electrolytes and some other potential stabilizers at different concentration on the calamine lotion formulas. They were also interested in the influence of pH on the stability of the lotion and if necessary to prepare a new formula. The U.S.P. XIII lotion, Stanko and DeKay's formula, Goldstein's formula, and a few others were included in this investigation.

The electrolytes studied were ammonium chloride, sodium citrate, potassium bitartrate, sodium phosphate, sodium salicylate, calcium chloride, sodium chloride, and alum. These were used in conjunction with the lotions in varying concentrations. They also studied variations of gums and wetting agents combined with the lotions. The results of this study showed that U.S.P. XIII calamine lotion containing 3% potassium bitartrate is the best lotion. This was followed by the U.S.P. XIII lotion prepared without lime water with Veegum as the suspending agent and containing 1% sodium phosphate. The third best lotion studied was U.S.P. XIII plus 1% ammonium chloride.

Blaug (31) continued the work of Escabi and DeKay and submitted a formula as follows:

I. Calamine	80 Gm.
Zinc oxide	80 Gm.
Bentonite magma	250 ml.
Glycerin	20 ml.
Potassium bitartrate	10 Gm.
Purified water qs	1000 ml.

This formula is similar to the U.S.P. XV except that purified water is used in place of lime water. Escabi and DeKay recommended .3% potassium bitartrate, while Blaug's formula calls for 1%. His second formula was as follows:

II. Calamine	80 Gm.
Zinc oxide	80 Gm.
Cellosize (WP 4400)	10 Gm.
Propylene glycol	100 ml.
Potassium bitartrate	2 Gm.
Dioctyl sodium sulfo-succinate 1% soln.	25 ml.
Purified water qs to make about	700 ml. 1000 ml.

One hospital using these preparations reported that formula #1 was the better of the two preparations, as it has good suspension properties and has excellent coating properties.

This review as presented does not cover all of the publications on this widely discussed preparation of calamine lotion, but it has been compiled to show the wide thinking and useful research which has been conducted in an effort to improve this preparation. The dermatologists are demanding this preparation and it is highly efficient in the treatment of many skin disorders. It seems that one should be able to eventually prepare such a preparation with all of the requirements built into its structure as to make it a highly prized medicant for skin disorders.

There have been many additional changes made in the products on the market, notable of which is the incorporation of antihistamines in this preparation to enhance its value.

A conclusion to be drawn from this review is the posibility of still further research from the cosmetic angle in making a preparation not too drying yet of such a nature as to dry up the open blisters from ivy and sunburn dermatitis, yet keeping the skin soft so that it does not further crack in the clearing process. More work is being done in our laboratories with this end in view.

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Chemical structure and antimicrobial activity of Bis-phenols

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The marked antimicrobial properties of 2,2′-methylenebis [4-chlorophenol], (G-4®, dichlorophene) and of 2,2′-methylenebis-[3,4,6-trichlorophenol], (G-11®, hexachlorophene) reported from our laboratories (1-4) induced the senior author to prepare numerous bis-phenols during the past two decades. This was done with the hope of finding more potent antiseptics, especially as additives for soap, and of correlating biological activity with chemical structure. By establishing such a relationship, a pattern might evolve which would lead to the synthesis of compounds with superior qualities.

The behavior of various series of bis-phenols against fungi, such as *Chaetomium globosum* and *Aspergillus niger*, had been thoroughly investigated by Marsh and his co-workers (5, 6). We shall present here only the study of the activity of a selected group of bis-phenols against *Staphylococcus aureus* and *Escherichia coli*, and against a pathogenic fungus, *Trichophyton mentagrophytes*, and their comparison with hexachlorophene being chosen as a standard in view of its wide use as an antiseptic. It had been previously stated that bis-phenols, such as hexachlorophene or bithionol, do not kill bacteria rapidly whether they are on the skin or in any substrate, but exert a slow inhibitory action which in time will lead to the death of the organisms. For this reason, we determined the bacteriostatic levels of the compounds against the three microorganisms employing serial dilution technique. The results are shown in Tables 1 to 5. While, naturally, we have done work of this kind in previous years, the data presented here are derived from a recent study carried out under identical conditions. *S. aureus* would appear to be the most suitable organism for the evaluation of topically applied antiseptics; we felt that the tests with this organism should also be run in the presence of soap. The latter results provide a better indication of the utility of the compounds, especially as the principal use of active bis-phenols lies in the field of antiseptic and deodorant soaps and detergents. We are aware that the data obtained *in vitro*, while helpful for selecting the most promising compounds, do not constitute the final criterion of their value as skin antiseptics. This depends on *in vivo* studies of their ability to degerm the skin, as may be demonstrated by standard hand-washing techniques. There appears to be a fairly good relationship between *in vitro* and *in vivo* activity (7).

* *Many references are found in the review article "The Bis-Phenols" (4).*

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Experimental

Preparation of the Bis-Phenols

The compounds used in this study were prepared by standard methods. Many of the compounds listed in the tables have been described in the scientific literature and in patents;* for the substances which are new, the synthesis is largely evident from their structure and the procedure may be derived from the known preparation of analogs. This paper would not be the proper place to present experimental details; it may suffice to make a few general statements.

All compounds of Table 1 are derivatives of diphenylmethane and were obtained by condensation of the appropriate phenols and aqueous formaldehyde or paraformaldehyde in the presence of sulfuric acid of varying strength, the conditions depending on the reactivity of the phenol employed. The asymmetrical compounds Nos. 13 and 14 had to be made in two steps: alkaline condensation of 2,4-dichlorophenol with formaldehyde yielded 2,4-dichloro-6-hydroxymethylphenol which was brought to reaction with *p*-chlorophenol (No. 13) and 2,4,5-trichlorophenol (No. 14) under acid conditions. It should be mentioned that compound 9 was synthesized by debromination of compound 5 by means of zinc dust in potassium hydroxide solution.

Similarly, the compounds listed in Table 2 were prepared by condensation of the monophenols with acetaldehyde, chloral, benzaldehyde and substituted benzaldehydes. Bromination of compounds 15 and 18 resulted in compounds 16 and 19, respectively. The stilbenediols (Nos. 20 and 21) were obtained from the corresponding benzoins (8).

The reaction of phenols with sulfur dichloride led to the formation of the 2,2′-thiobis-phenols listed in Table 3. If the phenols are substituted by more than one chlorine, aluminum chloride is needed as a catalyst. Dechlorination of compound 30 led to compound 29, bromination to compound 33.

The bis-phenols listed in Tables 4 and 5 were generally prepared by the same methods as had been employed for the compounds of the preceding tables. We might add that compound 43 was obtained by first preparing octachlorodiphenylene dioxide from potassium pentachlorophenoxide at 300°C., and heating the dioxide with sodium hydroxide in aqueous

methanol at 160°C. Compounds 45, 58 and 59 resulted from the oxidation of the corresponding 2,2′-thiobisphenols with hydrogen peroxide. Bromination of compound 46 let to compound 47, chlorination of compound 48 to compound 49.

Antimicrobial In Vitro Study

Bacteriostatic and fungistatic levels were ascertained for *Staphylococcus aureus* ATCC 6358, *Escherichia coli* ATCC 11229, and *Trichophyton mentagrophytes* ATCC 9129 by serial dilution technique. Stock solutions of the compounds to be tested were made in acetone or N,N-dimethylformamide followed by twofold serial dilutions in alcohol (S.D.A. 30). Aliquots from the solvent serial dilutions were added to A.O.A.C. agar tubes for bacterial tests and Sabouraud's Dextrose Agar (Difco) tubes for fungal tests. The tube contents were mixed and poured into sterile plates which were then spot-inoculated with the test organisms. Twenty-four hour A.O.A.C. broth cultures of *S. aureus* and *E. coli* were employed, the inoculum being diluted 1-100 in A.O.A.C. broth. Spores and mycelia fragments harvested from a seven-day Sabouraud's dextrose agar slant with water served as the Trichophyton inoculum. Inhibitory levels were recorded at the end of forty-eight hours at 37° C., and seven days at 30° C., for the bacteria and mold respectively. Controls were included to demonstrate lack of inhibition by the solvents employed. Replicate tests were conducted on different days in an attempt to minimize the day-to-day variation in the test procedure.

Bacteriostatic levels of the compounds in the presence of soap were ascertained by a similar technique except that aliquots of the stock solutions were added to a Maxine (Swift & Co.) soap solution. Twofold serial dilutions were made in sterile tap water and aliquots of the dilution series added to A.O.A.C. agar. Plates were streaked with *S. aureus* as previously mentioned, incubated at 37°C., and results recorded at the end of forty-eight hours. Controls demonstrating lack of inhibition due to the soap were employed.

Table 1—Antimicrobial Activity of 2,2′-Methylenebis-Phenols

No.	Compound Substituents	Minimum Inhibitory Level, μg./ml. *S. aureus*	*S. aureus* (in Presence of Soap)	*E. coli*	*T. mentagrophytes*
1	3,4,6-Trichloro,hexachlorophene, G-11®	0.39	1.56	25	3.12
2	4,5,6-Trichloro	0.39	0.78	25	6.12
3	3,4,5-Trichloro	0.20	0.39	6.25	3.12
4	4-Bromo-3,6-dichloro	1.56	6.25	50	6.25
5	4-Bromo-5,6-dichloro	1.56	6.25	>50	12.5
6	4-Chloro, dichlorophene, G-4	3.12	12.5	25	6.25
7	4-Methyl	25	>12.5	>50	50
8	4,6-Dichloro	0.78	1.56	>50	1.56
9	5,6-Dichloro	3.12	6.25	>50	3.12
10	4,6-Dibromo	0.78	3.12	>50	3.12
11	6-Bromo-4-chloro	0.78	1.56	>50	6.25
12	4-Chloro-6-methyl	1.56	3.12	>50	6.25
13	2,2′-Dihydroxy-3,5,5′-trichlorodiphenylmethane	1.56	3.12	50	3.12
14	2,2′-Dihydroxy-3,5,3′,5′,6′-pentachlorodiphenylmethane	0.78	1.56	50	3.12

Table 2—Antimicrobial Activity of 2,2′-Ethylidene and 2,2′-Benzylidenebis-Phenols, and of 2,2′-Stilbenediols

No.	Compound	Minimum Inhibitory Level, μg./ml. *S. aureus*	*S. aureus* (in Presence of Soap)	*E. coli*	*T. mentagrophytes*
	2,2′-Ethylidenebis (. . .phenol) Substituent				
15	4-Chloro	3.12	12.5	50	3.12
16	6-Bromo-4-chloro	3.12	6.25	50	3.12
17	4-Methyl	12.5	>12.5	>50	50
18	2,2′-(2,2,2-Trichloroethylidene)-bis(4-chlorophenol)	0.78	12.5	>50	3.12
19	2,2′-(2,2,2-Trichloroethylidene)-bis(6-bromo-4-chlorophenol)	0.78	12.5	>50	3.12
20	5,5′-Dichloro-2,2′-stilbenediol	3.12	12.5	>50	3.12
21	3,5,3′,5′-Tetrachloro-2,2′-stilbenediol	0.39	3.12	>50	6.25
22	2,2′-Benzylidenebis(4-chlorophenol)	3.12	>12.5	>50	>50
23	2,2′-Benzylidenebis(6-bromo-4-chlorophenol)	6.25	>12.5	>50	>50
24	2,2′-Benzylidenebis(4,6-dimethylphenol)	6.25	>12.5	>50	>50
25	4,4′-Benzylidenedithymol	1.56	12.5	>50	>50
26	4,4′-(2-Chlorobenzylidene)dithymol	0.78	6.25	>50	>50
27	4,4′-(4-Chlorobenzylidene)dithymol	0.78	6.25	>50	50
28	4,4′-(4-Hydroxy-3-methoxybenzylidene)-dithymol	3.12	6.25	>50	50

Table 3—Antimicrobial Activity of 2,2′-Thiobis-Phenols

No.	Compound	Minimum Inhibitory Level, μg./ml. *S. aureus*	*S. aureus* (in Presence of Soap)	*E. coli*	*T. mentagrophytes*
	Substituents				
29	...	>50	...	>50	25
30	4-Chloro	3.12	12.5	25	3.12
31	4-Bromo	3.12	12.5	25	3.12
32	4,6-Dichloro,(bithionol)	1.56	3.12	>50	0.78
33	6-Bromo-4-chloro	1.56	3.12	>50	1.56
34	3,4,6-Trichloro	0.78	6.25	>50	50
35	Tetrachloro	0.78	12.5	>50	>50
36	4-Chloro-6-isopropy-3-methyl	3.12	>12.5	>50	>50
37	4-Chloro-3,5-dimethyl	6.25	>12.5	>50	>50
38	2-Hydroxy-2-methoxy-5,5′-dichlorodiphenylsulfide	6.25	>12.5	>50	12.5

Table 4—Antimicrobial Activity of Related Bis-Phenols

No.	Compound	Minimum Inhibitory Level, μg./ml. *S. aureus*	*S. aureus* (in Presence of Soap)	*E. coli*	*T. mentagrophytes*
39	1,1′-Methylenebis(2-naphthol)	6.25	12.5	>50	6.25
40	3,3′-Methylenebis(2,4,6-trichlorophenol)	6.25	12.5	>50	3.12
41	3,3′-Methylenebis(4-bromo-2,6-dichlorophenol)	12.5	>12.5	>50	12.5
42	4,4′-Methylenebis(2,3,6-trichlorophenol)	12.5	>12.5	>50	3.12
43	2,2′-Oxybis(tetrachlorophenol)	0.78	1.56	>50	50
44	4,4′-Thiodiphenol	>50	...	>50	25
45	2,2′-Sulfinylbis(3,4,6-trichlorophenol)	12.5	>12.5	>50	25
46	*o,o′*-Diphenol	>50	...	>50	50
47	4,4′,6,6′-Tetrabromo-*o,o′*-diphenol	1.56	3.12	>50	6.25
48	Salicil(2,2′-dihydroxybenzil)	>50	...	>50	50
49	5,5′-Dichlorosalicil	3.12	12.5	>50	1.56

All bacteriostatic tests in the presence of soap were conducted at a soap/compound ratio of 50/1.

The values listed in Tables 1 to 5 are the higher numerical figures (μg./ml.) demonstrating inhibition in the twofold serial dilution series. If, e.g., one test showed inhibition at 3.12 μg./ml. and a second one at 6.25 μg./ml., the latter figure was chosen.

The relationship of the antimicrobial activity of bis-phenols to their structure had been studied previously. Bechhold and Ehrlich (9) were the first ones to demonstrate that halogenation of *o,o′*-diphenol enhanced its antibacterial potency. From the figures presented in our tables, it may be seen that halogen substitution in the rings is essential. Marsh and Butler (5) and Marsh, Butler and Clark (6), in their investigations on the fungicidal properties of bis-phenols, have discussed the dependence on certain structural features for high activity. Corey and Shirk (10) reported on the influences of chemical structure of a number of bis-phenols with an unsubstituted methylene bridge upon antifungal potency. From these and other papers, it is apparent that there is no correlation between bacteriostatic and fungistatic properties. Pfleger and his associates (11) investigated the *in vitro* antimicrobial activity of a series of bis-phenols with –S–, –SO–, $–SO_2–$, –S–S–, $–S_3–$ and $–S_4–$ linkages mostly in the ortho positions to the hydroxyl groups. The organisms employed were *Staphyloccus aureus*, *Salmonella paratyphi*, *Trichophyton gypseum* and *Torulopsis minor*. The authors stated, as we and others have also observed, that the 2,2′-thiobis-phenols are more bacteriostatic and fungistatic than the 4,4′-thiobis-phenols, and that the introduction of chlorine or bromine increased the antimicrobial activity considerably. None of the compounds tested inhibited *S. paratyphi* at a concentration lower than 12 μg./ml., whereas a number of compounds were active against *T. gypseum* and *S. aureus* at a level of

Table 5—Bis-Phenols Showing Inhibitory Levels of 50 μg./ml. Against the Three Test Organisms

No.	Compound
50	2,2′-Methylenediphenol
51	2,2′-Methylenebis(4,6-dimethylphenol)
52	2,2′-Methylenebis(6-bromo-4-methylphenol)
53	2,2′-Methylenebis(4-nitrophenol)
54	2,2′-Methylenebis(4,6-dichlororesorcinol)
55	2,2′-Methylenebis(3,5,6-trichloroanisole)
56	4,4′-Methylenebis(2,6-dibromophenol)
57	2,2′-Thiobis(4-chloroanisole)
58	2,2′-Sulfinylbis(4-chlorophenol)
59	2,2′-Sulfonylbis(4-chlorophenol)
60	2,2′-Dihydroxy-5,5′-dichlorobenzophenone
61	1,1′-Methylenebis(6-bromo-2-naphthol)
62	1,1′-Methylenebis(3,6-dibromo-2-naphthol)
63	4,4′-(3,4-Dimethoxybenzylidene)dithymol
64	Bis(3,5-dichloro-2-hydroxybenzyl)amine

1 μg./ml. and less. The most potent of these compounds against these two organisms and against *Torulopsis minor* was 2,2′-thiobis(4,6-dichlorophenol). A series of 2,2′-methylenebis-phenols, 2,2′-ethylidene and 2,2′-benezylidenebis-phenols, and a number of other compounds were examined by Florestano and Bahler (12) for antibacterial activity *in vitro* against a variety of organisms. They found that the compounds showed more or less specificity against gram-positive bacteria, being distinctly more active against them than against the gram-negative species tested. Halogen substituents were essential for potency; bromine rather than chlorine substitution resulted in greater activity against gram-positive organisms, while antibacterial action on the gram-negatives remained the same. Florestano and Bahler also stressed the significance of the position of the hydroxy groups in the rings.

Wendel (13) examined five bis-phenols, Nos. 1, 6 and 8 of Table 1, No. 40 of Table 4 and 4,4′-methylenebis (3,5-dichlorophenol), for their antibacterial potency against *S. aureus*, *S. albus* and a Streptococcus. He confirmed previous findings, namely, that the bacterial activity is connected with the number of chlorine atoms in the rings, an increase being shown from two to four to six chlorines; and that this enhancement is only evident if the hydoxyl groups are adjacent to the methylene bridge.

From the data presented in Tables 1 to 5 we are able to draw certain general conclusions (some of them in conformity with the findings of others) with regard to the relationship of the antimicrobial properties of the bis-phenols and their chemical stucture. However, before doing so, we would like to call attention to the two compounds most closely related to hexachlorophene, namely Nos. 2 and 3. No. 2, 2,2′-methylenebis-(4,5,6-trichlorophenol), demonstrated approximately the same activity as hexachlorophene being perhaps slightly more inhibitory against *S. aureus* in the presence of soap. No. 3, 2,2′-methylenebis (3,4,5-trichlorophenol), was outstanding, not only on account of its activity against *S. aureus*, but also because it was the only bis-phenol which showed a fair degree of bacteriostatis against gram-negative organisms. We have given its activity against *E. coli* as 6.25 μg./ml. This value resulted from two tests; in two other ones, the inhibitory level was found to be as low as 3.12 μg./ml. *Proteus vulgaris* and *S. typhosa* were inhibited at 6.25 μg./ml., *Pseudomonas aeruginosa* at 12.5 μg./ml. The question might be raised as to why compound 3, being definitely more active than hexachlorophene, at least *in vitro*, is not being studied *in vivo* and perhaps used commercially? The reason lies in the difficulty of economically making 3,4,5-trichlorophenol which is obtained in a multistep operation from *p*-nitroaniline (14, 15) and which is needed for the preparation of compound 3.

The following general statements may be made:

1. In general the antibacterial activity of the bis-phenols is rather specific, being stronger against gram-positive organisms than against gram-negative ones. However, compounds 1, 2, 3, 6, 30 and 31 are exceptions and do show good activity against *E. coli*. The presence of soap always lowers the inhibitory level against *S. aureus*. The potency against *Trichophyton mentagrophytes* is not striking, only three compounds (Nos. 8, 32 and 33) which have a similar chemical structure are fungistatic at a level lower than 2 μg./ml.

2. Linkage in the 2,2′-positions is essential for maximum activity. Previous observations (4) that bis-phenols linked in the 4,4′-positions are weak are generally correct; however, we observed that compounds obtained by the condensation of thymol with benzaldehyde and particularly with *o*- and *p*-chlorobenzaldehyde are quite active against *S. aureus*. However, in the presence of soap, the decrease in potency is more pronounced than with the 2,2′-methylenebis-phenols.

3. The methylene-bridged phenols are superior to the ones with ethylidene, benzylidene and thio-linkages in regard to activity against *S. aureus* in the presence of soap.

4. No compound without halogen in the phenol rings is active, with the exception of bis-phenols derived from thymol (Nos. 25, 26, 27), and one halogen must be in the para position. Chlorine substitution is usually preferable to bromine which is contrary to Florestano's findings (12).

5. Oxygen-containing linkages, such as –CO–, –SO–, $–SO_2–$, are detrimental.

6. Activity is lost if the hydroxyl groups are etherified (Nos. 55, 57). Esters, such as the diacetates, have been reported by Pfleger *et al.* (11) to be active. We found this to be true, and believe, in agreement with Pfleger, that the esters are hydrolyzed to a large degree to the free phenols during the tests.

7. Replacement of the phenols by naphthols (Nos. 39, 61, 62) leads to inactive compounds. The bis-compound from dichloresorcinol (No. 54) was also inactive at a level of 50 μg./ml.

Summary

Bacteriostatic and fungistatic data on a number of bis-phenols have been presented and the relationship between chemical structure and antimicrobial activity has been discussed. The standard of reference was hexachlorophene which was not surpassed in bacteriostatic activity against *S. aureus* and *E. coli.*, except by one of its isomers, 2,2′-methylenebis (3,4,5-trichlorophenol). The latter compound is distinctly more potent, but its manufacture would be difficult and costly.

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Oligodynamic silver and its compounds as used in cosmetics

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Herodotus tells in his stories that Cyrus, king of Persia drank only water of the Coaspe River, boiled and conserved in big silver mugs, which he transported with him in all his war expeditions.

Therefore, he was an instinctive precursor of modern experimental hygiene. The water was conserved pure after contact with the silver walls of the receptacle, through which the ionization of the metal exercised a bacteriostatic and bactericidal power on the germs which live and develop in water.

Thanks to his scientific knowledge, the human being has discerned the bactericidal power of silver. He did study its mechanism and did research the ways to carry on industrially this virtue in order to resolve the sterilization problems. The microbicidal power of the ionized silver on the lower organisms depends from a physical chemical activity, very complex, which includes:

1. A physical action of reciprocal attraction, after the electric effect between the positive ions and the live cells, which have to be attacked.
2. A catalytic oxidation action exercised by the silver oxide present.
3. A penetration by osmosis of the ionized metal in the interior of the attacked cells, with a consequent formation of metallic protein, toxic to the lower animals.

The reason for this toxicity, exercised by the ionized silver on the microorganisms, can be attributed to the fact that in nature large quantities of other numerous ionized metals are found: iron, aluminum, sodium and of this omnipresence, their inoffensivity is justly derived. On the other hand, silver is never present in dispersed ionic form, because the minerals which contain it (galena, quartz, etc.) are practically insoluble and nonionizable in water. In consequence, there is no immunity to the oligodynamic silver by the microorganisms. The first modern oligodynamic silver applications were limited to the simple contact effect, that is to say that ceramic carriers were endowed with a very thin silver pellicle or contained silvered porcelain links. The sterilizing effect was very slow, about 10 hours contact and in consequence, the practical application was limited to small quantities of water.

Afterwards, Kreuze and von Nageli, created the electrolytic dissociation system of silver by continuous current at feeble potential on pure silver electrodes, a system which was called the oligodynamic Katadin method. The production machine was called electrostimulator and found immediate practical applications in the treatment of water of potable quality.

The fundamental principle of the Katadin method is the oligodynamic sterilizing action of the ionized groups of the very finely divided collodial silver.

In practice, the liquid to be sterilized went through a receptacle in which two silver electrodes were conveniently placed between which a very weak current was passing. Very feeble colloidal or semi-molecular silver traces passed into the liquid and as these very finely divided particles exercise enormous surface tensions; the organisms present are killed. It is generally admitted that the silver proportion passing into solution is less than 0.01%.

This process is used in West-Germany for water sterilization. The Ag ion has a sterilized action on numerous bacteria gram negative; it can also be utilized to treat water in which "salmonella" have to be suppressed.

The way of oligodynamic sterilization with the Katadin stimulators, presents equally some practical inconveniences, which limit and in some cases void the positive effects.

A thorough study of the method demonstrated that the bactericidal action of the electrolytically dissociated silver depends of the corpuscular fineness of the dispersion.

The quality of the water to be sterilized from the physical and chemical point of view is most important. The dispersion of silver and organic matter cells for precise dosing of silver, because the colloidal organic dispersions absorb the ionized metallic silver.

The alkaline-earth salts (calcium, magnesium) which determine the hardness of the water, are in part, precipitated by the electric effect and, being combined with the ionized dispersion, provoke a coagulation

which impoverishes the liquid and its metallic contents. The electrodes are endowed with a compact coat of precipitated salts which reduce the effective surface and the electrolytic dissociation, modifying consequently the degree of fineness of the dissociated metal.

The ionized silver easily combines with other chemical elements to form neutral salts as by-products of the sterilization.

The chlorides yield chlorine in forming silver chloride which is almost entirely inactive.

The formation of silver chloride creates in the liquid an opalescent dispersion with noxious effects.

The H_2S also very often present in shallow and deep waters, combines with the ionized silver giving silver sulphide which is absolutely neutral from the point of view of the sterilization action.

Considering the very small silver quantities needed, minute traces of H_2S can nullify totally its bactericidal action.

Considering all these inconveniences, other research was undertaken to modify the method and to eliminate entirely the negative causes, (Conconi, Bertarelli, Peragallo, Fiori, Pauli).

These researchers did ascertain that the oligodynamic activity is proportionate to the present oxide on the metal.

They studied several oxidation methods to realize this condition and to obtain an inert silver compound at each new chemical combination, not attacked by the H_2S, dissolved in water in remarkable quantities and therefore infinitely more active. After the success of their research, they put to disposition an oligodynamic silver which can be applied to a new series of products with new hygiene characteristics. This silver appears either dissolved in water with high ionized concentration of silver (50,000 gamma/litre and more under a very fine stabilized dispersion form) or in dry powder form. This activated water, thanks to its important characteristics of permanent activity, can be employed in numerous uses and marks real progress in hygiene and sterilization through oligodynamic silver.

This new way of processing of oligodynamic ionization is a method for the ancient specific applications of biological depuration of the water: nourishing use, sterilization of pools, production of sterile ice etc. . .

The only difference consists in the fact that instead of passing the liquid to be sterilized through a recipient of silver electrodes, the ionized water with high concentration is introduced into this liquid.

But the new method is the only one which allows new interesting uses in the hygiene branch.

The ionized water with high concentration has, in fact, the characteristic of penetrating the interior of microporous bodies and depositing there, through an osmotic process, the metallic ionized dispersion. These bodies, in the active solution, acquire sterilized and bactericidal properties.

This phenomenon was studied by Pilod, Codvelle, Lakhovsky, Lasseur and Vernier. This ionized penetration can be compared to a dyeing, because the fineness of dispersion and the characteristic adhesion of the metal have a conduct very similar to that of a good dyestuff. Experimental tests were made with very diverse materials, treated by immersion, hot or cold, in the ionized liquid at high concentration. In the cosmetics branch, this method was applied with a slight difference. Water, generally always present in small or large quantities in beauty products under non powdery form, was partially replaced with high ionized silver concentration water, at a level up to 1000 gamma of silver per kg or litre of finished product.

We ascertained the following:

1. The product so treated does not present any traces of toxicity, nor of irritation, because the silver quantities are insignificant.
2. Its organoleptic properties (odor, flavor, color) do not undergo any modification.
3. It possesses a disinfectant antimicrobial power, active against pathogenic, Micromycetes, fungi, etc. Consequently, it preserves itself indefinitely and acts as a biologically active agent in some skin infections.
4. It possesses a deodorizing power.

Concerning point 1 (non toxicity of the treated product) which is particularly important, we cite the following details written by Dr. Arquie from Paris: "The residual silver, in sterilization by silver ions, is insoluble. It is the insoluble silver ions which are bactericidal" and "it is well established that they are devoid of all toxicity" to the organism. In fact, Hazard, in his Summary of Therapeutics and Pharmacology (ed. 1950) states precisely:

"Metallic silver, being insoluble in hydrochloric acid at ordinary temperature, cannot be reabsorbed by the digestive tract and must therefore be considered as void of toxic properties".

Soluble silver will, in some cases, give argyry phenomenons. But to do this, silver dosages must reach 1 g for 100 g weight of the animals.

Beauty products in which this new technic gives more than satisfactory results in transforming them into veritable biological deodorizers, are the following:

- Disinfecting soaps, tonics, and creams for oily, acne and pimpled skins.
- Lotions against falling hair (often due to the presence of microorganisms).
- Talc for babies where the water at high concentration is replaced with powder obtained from the same water.

The very low cost, the unlimited duration of its activity, and the facility of its use, caused us to make use of this water at high ionized silver concentration, which was called Silvercon.

In order to obtain an inert compound at each new chemical combination that was not attacked by H_2S, other seekers directed their searches to silver compounds such as Silver Benzene Sulfinate; another one was a complex by the reaction of silver chloride with ethylene thiourea (Schimmel briefs no. 296, November 1959), and silver protein (US 2.893.918 Abramson, Harold A. July 1959). The silver protein is most commonly used as a deodorizing agent.

Several forms of silver protein are commercially available. The mild silver protein of the National Formulary is sold under various trade names, e.g. Argyrol (Barnes) and Silvol (Parke, Davis). This type of colloidal silver may be prepared from silver oxide

and gelatin in the presence of alkali, or from silver oxide and serum albumin. Mild silver protein contains around 20% silver; it occurs in the form of dark brown scales or granules which dissolve freely in water forming a colloidal solution. There are very few silver ions present in the solution as shown by the fact that it does not give a precipitate with dilute solutions of sodium chloride.

Colloidal silver iodide, N.F., contains approximately 20% of silver iodide in a gelatin base. It is sold under the name Neo-Silvol (Parke, Davis). This form of silver protein is preferred for use in cosmetic deodorants because of its light color. It is a pale yellow granular solid and forms solutions which have an opalescent color.

The antiseptic action of colloidal silver prevents infection of small abrasions caused by shaving. The astringent lotions containing silver protein are useful not only as cosmetic deodorants but as deodorizing and antiseptic lotions for the treatment of athlete's foot.

Microscopic examination of hair in normal and polarized light

by Dr. F. Keil

Summary by the author of a talk with slides held on February 6, 1960 in Hamburg at the meeting of the Society of German Cosmetic Chemists

The speaker first explained the apparatus used and the method of taking pictures. By means of some hair root specimens the differences in color and the reproduction of details with the use of the same apparatus but different exposures was demonstrated. Pictures were made in light and dark field with normal, unpolarized light, with phase contrast optic and with polarized light. The advantages of the use of polarized light were explained and the influence of different imbedding fluids with different refractive indexes was discussed. The making of hair cross-section by a fast method was explained. This consists in the imbedding of a hair sample between two transparent celluloid plates which previously have been moistened with acetone. After only a few minutes they can be installed into a microtome with the help of blocks of hard paraffin and then cut. The behavior of hair in different liquids was also shown by means of micro-pictures. The change in polarization color connected with this was also discussed.

The hair samples themselves were glued to the object carrier in little loops with a diameter of a few millimeters which made it possible to treat just a little piece of the hair with liquid and thus to avoid a swelling longitudinally. This prevents a dislocation of the specimen during the test and enables the operator to relocate the treated spot even after several days. Pictures of specimens in distilled water phenol, concentrated HCl, concentrated H_2SO_4, conc. HNO_3, conc. NH_4OH, conc. KOH, in bleaching and in cold wave fluids were shown. It was shown that the attack on the hair even if the curvature is slight takes place faster on the concave side. The speaker pointed out that the polarization apparatus gives us the possibility to observe the speed of penetration of fluids into the hair, to locate faults in its structure and to observe the processes taking place when hair is chemically treated. Hair after all is not only an organic chemical protein compound but has at the same time a distinct structure which we would like to know more precisely.

What makes a flavor?

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Ask 100 people why they like Blue Cheese and you'll get 100 answers. As with any other food product that might be selected, some *won't* like it. Others will name its good taste, or its good flavor. And not one in the entire 100 will really know.

Dr. Robert Coffman of the Food Research section at General Mills' Central Research Laboratories is a member of a pioneering group that for the past three years has made a science of learning about food flavors through chemical analysis.* Recently they tested the hyper sensitivity of a special, laboratory-built, gas-liquid chromatograph by launching an investigation of the composition of the volatile substances that combine to make up the "natural" flavor of Blue Cheese. Infrared spectroscopy aided the group in identifying isolated components.

It was found that a total of 29 neutral volatile substances were present in this flavor along with about 18 acids, of which 10 were present in major quantity. The main neutral components were found to be straight chain methyl ketones containing five to eleven carbon atoms. Each one of them was accountable for a portion of the blue cheese odor. The 7 carbon ketones accounted for more than 50 per cent of the neutral volatiles. The one most prominent acid was capric.

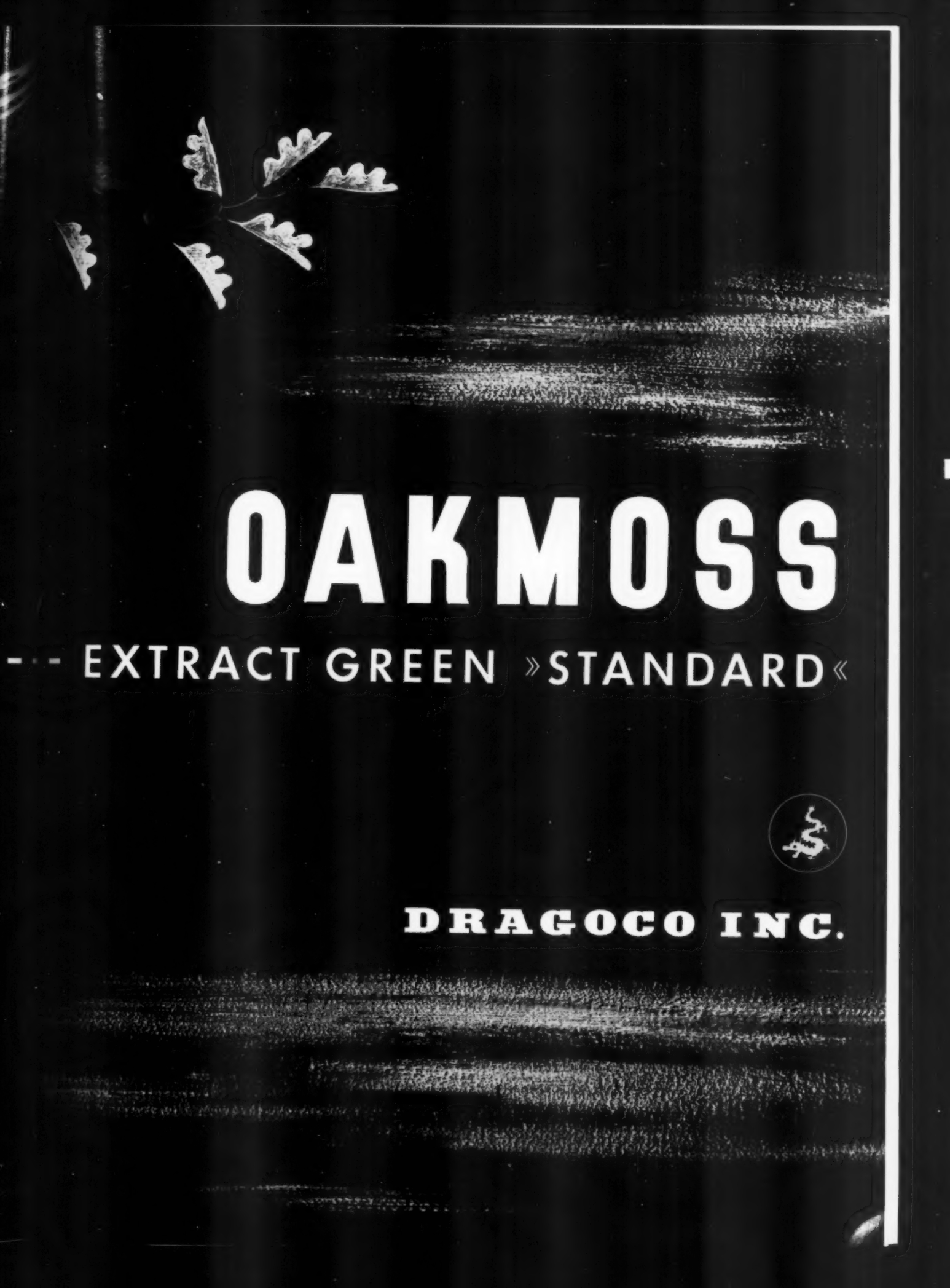
Once the identifications were made, the Food Research group reversed the procedure. They began with the ketones and acids and created a synthetic Blue Cheese flavor that came very, very close to the "natural" flavor.

Here, a relatively simple flavor had been broken down into its separate fractions. The findings in general bore out published chemical analyses obtained by other scientists in analyzing Blue Cheese flavor. But some new information had been gained, also.

Perhaps it's not important that a consumer know exactly why he likes a certain food flavor. But because we are all creatures of habit and tend to eat the same foods again and again, it *is* important to a food processor. Flavor can determine whether a product will be a success or failure.

Small wonder, then, that today there is much research going on in the food industry in connection with flavors. And that every bit of information that can be gained is vital.

**See Progress Thru Research, Vol. 11, No. 1, 1957 and Vol. 12, No. 1, 1958*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Flow properties of dry particulates

BY E. J. CROSBY
Department of Chemical Engineering
The University of Wisconsin

The laws which govern the behavior of solid particulates deviate markedly from those which describe the characteristic behaviors of perfect fluids and solids. Only for the extreme cases of very fine powders and well adhered masses do particulates exhibit properties comparable with those of these fluids and solids. Even these comparisons are limited to very simple considerations. Neumann (24) has discussed in some detail the limitations of drawing rheological analogies between particulate systems and well-known fluid and solid systems.

There has been a great deal of interest in the flow of solid particulates, but very little theory has been developed to describe the process. Further, little attempt has been made to separate the many variables which seem to affect the flow phenomenon. This latter situation may exist, in part, because of the experimental difficulties involved. Nevertheless, efforts to empirically correlate laboratory results have been made and these appear to be successful in a limited sense.

Although the behavior of static systems is not commensurate with that of dynamic systems, it seems to merit consideration. It has been observed that the flow characteristics of particulates are, in certain ways, related to the properties of static beds.

Further, distributions of particle sizes are always encountered in particulate systems. It is helpful to study uniformly sized particles in order to better understand what occurs within a system containing a wide distribution of particle sizes.

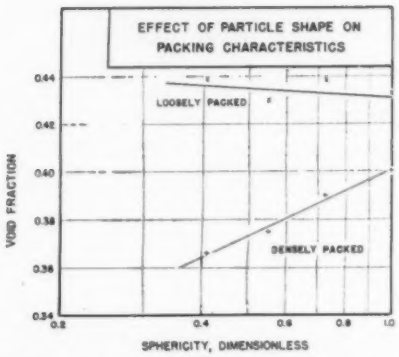
Packing Characteristics of Uniformly Sized Particles

In considering the characteristics of uniformly sized particles it is most convenient to analyze the situation for spheres and then to extend the results to non-spherical particles. A great many particulate systems take on approximately spherical dimensions; the major difference being in the nature of the particles' surfaces.

Spheres can be packed in six different ordered arrangements and the properties of these packings have been calculated (9, 20). These calculations show the minimum void fraction for spheres to be about 0.26. Randomness is the condition which exists in bulk materials and for spheres the void fraction is somewhat greater than 0.26. The void fraction obtained for spherical particles by prolonged shaking is about 0.37 to 0.40, depending upon particle density and surface characteristics, if the diameters of the spheres are relatively large. Theoretically the void fraction is independent of particle size. However, in practice, this fact has been found not to hold for the smaller particle sizes as numerous investigators have observed (2, 8, 27). Even though the void-fraction level and the particle size, where deviation from constant void fraction occurs, vary with the type of material, density and particle shape; the actions of the spheres are reproduced by the non-spherical particles.

A variety of explanations have been proposed as to why the void fraction increases with a decrease in particle size in the small size groups. It seems that, for particles of the same density and surface characteristics, the proximity of the particles are a function of the number of points of contact or surface area per unit mass per particle. When a mass of particulate matter is poured into a container a considerable amount of arching and bridging occurs, and upon vibration this arching and bridging is reduced. The larger particles will, when disturbed, exert greater forces than the smaller ones; however, the number of points of contact per particle, for any one type of packing, remains the same. For small particles, the number of contact points needed to maintain stability is then less than for large particles. Calculations show that the void fraction varies inversely with the number of contact points for ordered packings of spheres (20). A further factor in promoting increased voids with a decrease in particle size is the fact the settling rate of small particles is less than that for large particles. The fractional drag for single spheres in a fluid is directly proportional to surface area of the sphere, and the surface area to

Figure 1



weight ratio for a sphere is inversely proportional to the sphere's diameter.

Little conclusive information is available concerning the effect of particle shape upon packing. Goetzel's (17) review on this subject seems to indicate that the greater a particle's shape deviates from that of a sphere, the denser will be the packing under close packing conditions if the particle's surface is smooth. To test this idea four diversely shaped, approximately uniformly sized varieties of seed were analyzed with respect to their packing characteristics. The seeds were chosen such that the effects of particle size, density and surface roughness were minimized. The void fractions are correlated with the particles' sphericities, for both loose and close packing, in Figure 1. The sphericity of a particle is defined as the ratio of the surface area of a sphere having the same volume as the particle to the surface area of the particle (6). These results support Neumann's (24) contention that long needles and fibers can yield much lower voids than can the more symmetrically dimensioned particles.

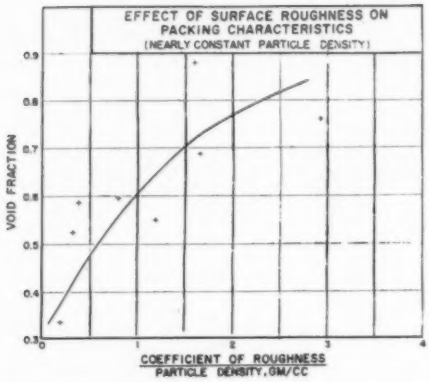
Inspection of available data indicates that particle density has little influence on voids. However, in the fine particle-size range, where the void fraction is no longer independent of particle size, the effect of increasing the density might be to reduce the void fraction.

The void fraction of a particulate system increases as the density of the medium, in which the particles exist, increases (13).

Any surface phenomenon which would promote difficulty in one particle sliding past another, such as surface roughness or cohesiveness, will promote greater voidage. For dry solids, the degree of surface roughness is an important consideration. Robertson and Emodi (26) proposed that the roughness of a particle's surface be characterized by a roughness coefficient. This coefficient may be defined as the ratio of total exterior surface of the particle to the gross exterior surface of the particle. Neumann (24) investigated a number of fairly uniformly sized powders and reported their void fractions for close packing as well as the total exterior surfaces per gram of powder. If it is assumed that the densities of the powders are about equal, roughness coefficients can be estimated from these data. The surface roughness is correlated with void fraction in Figure 2.

Separation of the effects of particle shape and surface roughness on the packing of particulate masses is

Figure 2



complicated by the problem of differentiating between variations of shape and roughness.

Packing Characteristics of Controlled Mixtures of Particles of Different Sizes

The majority of particulate systems are composed of a distribution of particle sizes. However, systems containing two, three and four size-components merit consideration. Furnas (15) and Fraser (12) have investigated the packing characteristics of binary systems. They found that the fraction of voids always decreased upon the addition of larger or smaller particles no matter what the size ratio between the particle diameters might be or how the masses of the two species were proportioned.

For systems of more than one component, in which the difference between particle sizes is such that no bed expansion is realized, the correct amounts of variously sized materials necessary to yield mixtures of maximum density are easily calculated (9). However, the condition of no bed expansion is not realized in practice. Furnas (16) has analyzed this situation and calculated the minimum expected voids for two, three and four component systems as a function of the ratio of smallest to largest particle size.

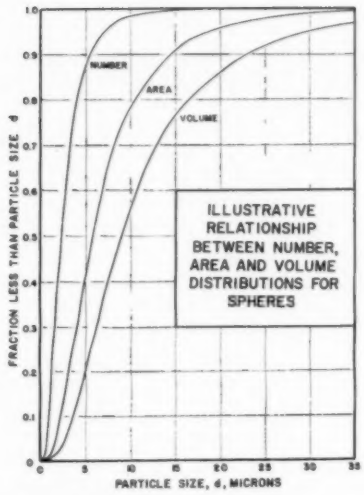
When a distribution of particle sizes is encountered, the relationships developed by Furnas are not applicable. Actual distributions composed of certain preselected, discrete size classes generally will not randomly pack in the desired manner to give minimum voids; and they will also generally segregate when disturbed. Stable systems, with minimum voids, seem to be those in which the number of particles in each size class increase as the particle size decreases (14). For these stable systems Andreasen and Andersen (2) have suggested a distribution law, which will produce the densest packing.

$$F\left(\frac{d}{d_m}\right) = \left(\frac{d}{d_m}\right)^q$$

where: $F(d/d_m)$ = cumulative weight fraction less than (d/d_m).
d = particle size
d_m = maximum particle size in the particulate system
q = distribution parameter

Experimental analysis of this law determined the values of the distribution parameter which would give minimum voids to be 1/2 for loose packing and 1/3 for dense packing. The resulting distributions agreed very well with the empirical Fuller Curve (14) which is used to formulate concrete mixes.

Figure 3



Packing Characteristics of Actual Distributions of Particles

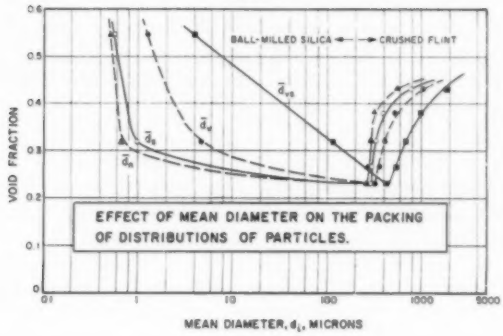
Actual distributions generally do not follow the laws necessary to produce masses with low voids. In fact, numerous different particle-size distributions, for any one material, will yield the same void fraction (12, 4).

Distributions which characterize a particulate mass are usually given in the form of the weight distribution; however, the bulking property of particles is dependent upon surface area as well as upon mass. Figure 3 shows the relationship between the number, surface and weight distribution for a particulate mass composed of spherical particles of uniform density. At any given size class the fraction of total area, resulting from all particles of smaller sizes, is greater than the fraction of total mass resulting from these same particles. Therefore, a small percentage of fines, on a weight basis, can conceivably have a marked effect upon the bulking characteristics of a particulate material.

In dealing with distributions of particles it is convenient to discuss or compare a mean particle-size with the property of interest instead of the entire distribution. The most common are the number-, surface- and volume-mean particle-sizes. In the case of mono-dispersed systems, the bulking properties appear to be related to the ratio of the particles' mass to surface-area. An extension of this conclusion to distributions of particles suggests that the bulking properties could be characterized by a hypothetical mean particle whose diameter is such that its surface to mass ratio is the same as that for the entire distribution of particles. Such a diameter is the volume-surface or Sauter mean diameter (28). A complete coverage of the statistics of particle-size distributions is given by Herdan (20).

Work (32) has reported data on the variation of bulk density of silica during a ball-milling operation. The void fraction decreased initially and then increased as comminution proceeded. Particle-size distribution data for only two of the six bulk density samples were reported. Combination of these data with those of Andreasen and Andersen (2) allows a comparison of the bulking tendency with a mean particle-size of the distribution. Figure 4 summarizes these

Figure 4


BALL-MILLED SILICA
CRUSHED FLINT
EFFECT OF MEAN DIAMETER ON THE PACKING OF DISTRIBUTIONS OF PARTICLES.
VOID FRACTION
MEAN DIAMETER, d_1, MICRONS

$\bar{d}_n$ = number mean diameter
$\bar{d}_s$ = surface mean diameter
$\bar{d}_v$ = volume mean diameter
$\bar{d}_{vs}$ = volume-surface or Sauter mean diameter
d_1 = $\bar{d}_n$, $\bar{d}_s$, $\bar{d}_v$ or $\bar{d}_{vs}$

data and indicates the Sauter-mean diameter to be the most satisfactory mean diameter for correlation. Certainly there are other parameters which should also be considered to accurately predict the bulking property of distributions of particles; for example, the maximum particle size.

Other Properties of Particulate Masses

Solid particulates exhibit certain angular properties which have been found to be related to the manner in which they flow. The three most important angular properties appear to be (i) the angle of internal friction, (ii) the angle of repose and (iii) the angle of wall friction. The dynamic forms of these angles are undoubtedly the most useful, but the static forms are those usually reported.

The angle of internal friction is probably the most characteristic angular property of particulate masses even though it is not often reported in the literature. Zenz (34) has extensively reviewed many of the numerous simple experimental techniques for measuring this angle. The most important factors influencing it are particle size, particle shape, surface roughness and, for heterogeneously-sized mixtures, the particle-size distribution.

The angle of repose is the most commonly measured and reported angular property. There are two static angles of repose; a drained angle and a poured angle. For systems made up of uniform particle sizes, both angles are the same. However, for a distribution of particle sizes, the drained angle is greater than the poured angle. This is because the smaller particles generally possess a larger angle of repose and therefore have a retarding effect on the flow of the larger particles. The exact opposite effect is produced for the poured angle. The same factors which influence the angle of internal friction also affect the angle of repose. Neumann (24) has shown the angle of repose to be related to the roughness of the particles' surfaces. The angles of repose discussed here are the static angles. Franklin and Johanson (11) have suggested a unique and simple method for measuring the dynamic angles.

McCabe and Smith (22) suggest that the difference between the angles of repose and internal friction is due to the difference in the packing of the material near the surface and that under the surface of a granular mass; the surface grains being more loosely packed

than those internally located. These two angles are expected to be the same for a truly homogeneous mass.

The static angle of wall friction is best obtained by measurement of static pressure in circular containers (21, 30).

It is a unique property of particulate masses, as compared with fluids, that the pressure at any point within a mass, which is at rest, is not the same in all directions. Pressures in all directions, other than that of the applied pressure, are less than the applied pressure and the minimum pressure is in a direction normal to the applied pressure. Further, applied shear stresses are transmitted throughout the static mass unless rupture occurs. The normal and applied pressures are related to the internal angle of friction in a simple manner (23).

It has been observed that the ratio of the normal pressure to the applied pressure is constant and is a characteristic of the particulate system. This ratio appears to be a function of the shape and the interlocking tendencies of the particles, the degree of packing and other particulate properties. It also appears to be independent of particle size except for very small particles. In this latter case, the normal pressure approaches the applied pressure as the particle size decreases.

Gravity Flow of Particulate Materials

It is well known that the static pressure exerted on the bottom of a chamber containing granular material is somewhat less than that exerted by an equal volume of liquid whose density is equivalent to the bulk density of the granuals.

Jannssen (21) developed a relationship to predict the pressure at the bottom of silos when they contained grain which is given below.

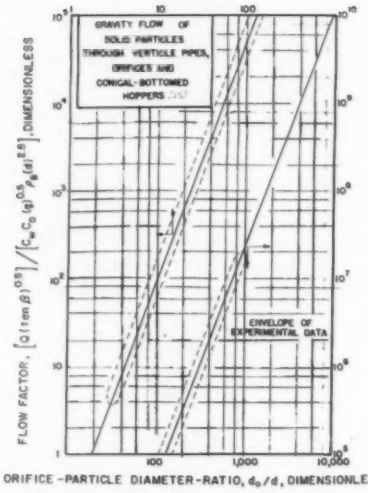
$$p_B = \frac{D\,\rho_B\,g}{4\,\mu\,k\,g_c}\left[1 - e^{\frac{-4\,\mu\,k\,h}{D}}\right]$$

where: p_B = pressure on the bottom of the silo.
D = diameter of the silo.
ρ_B = bulk density of the particulate material.
g = acceleration of gravity.
μ = tangent of the angle of wall friction (coefficient of surface friction).
k = ratio of the normal pressure to the applied pressure for the material being handled.
g_c = gravitational conversion factor.
h = height of material in the silo.
e = base to the natural logarithms.

Shaxby and Evans (30) found this same relationship to be equally applicable for powders in small cylindrical containers. Neither of these two expressions are exact descriptions of the actual situation but they are very close approximations. When the pressure exerted by the central core of a particulate bed is of interest, such as the static pressure over an orifice, the coefficient of internal friction for the particulate material should probably be used in the Jannssen equation rather than the coefficient of surface friction. The development of the Jannssen equation suggests that arching occurs in the silo. Vibration can prevent arching under flow conditions but can promote arching when there is no flow.

Numerous relationships have been developed to predict the flow rates of particulates but the majority are limited to specific materials and conditions. A

Figure 5



Q = solids flow rate, mass per unit time
β = static angle of repose
g = acceleration of gravity, length per unit time per unit time
ρ_B = bulk density of solids, mass per unit volume
d = particle diameter, unit length
d_o = orifice or pipe diameter, unit length
C_w = wall correction factor, dimensionless ($C_w = 1$ for d_o/d greater 20 to 30)
C_o = hopper-bottom correction factor, dimensionless ($C_o = 1$ for flat bottomed hoppers)
Note: All length and time units must be consistent.

rather extensive review of these relationships has been made by Zenz (35). Zenz concluded that the correlation by Rausch (25) was the most comprehensive and found that it agreed well with the data of three other investigators as well as those of his own. Rausch's correlation is summarized in Figure 5.

The relationship between the static-pressure expression of Jannssen and the correlation by Rausch is very close. It has been found that the pressure at the bottom of a tube, through which particulates are flowing, is only about 10 to 15 percent greater than that for static conditions. The applicability and use of the angle of repose rather than the angle of internal friction in Rausch's semi-empirical expression is probably a result of their near equality for many materials.

As might be expected, a wall effect is encountered when the ratio of the outlet or tube diameter to the particle diameter becomes too small; less than 20 to 30. This value is undoubtedly a function of particle shape and surface properties. A number of relationships have been developed to predict and account for this effect. Zenz has summarized some of these relationships along with certain experimental data in terms of a wall-effect correction. Franklin and Johanson (11) have presented data which indicate that the flow rate is also influenced by the ratio of the hopper diameter to the outlet diameter when this ratio becomes too small.

For those cases where hopper bottoms have inclined sides instead of flat bottoms, it has been found that the inclined sides give improved flow rates. A correction factor to account for this effect has also been presented by Zenz.

Unfortunately practically all flow-rate studies have been restricted to mono-dispersed systems or those with a very narrow size range. The correlation by

Rausch was based on these types of systems and may not be applicable to distributions of particles. However, as in the case of predicting the bulking properties of particulates, it may be possible to define a characteristic diameter, or some other parameter of the distribution, which will allow the correlations for single sizes to be applicable to distributions.

Other Characteristics of Flowing Particulates

Water of hydration or equilibrium moisture cannot be discounted when considering dry particulates. This small amount of moisture, coupled with surface roughness and particle shape, manifests itself in "sticky" particulate masses when very small particle sizes are encountered. It has been suggested that particles under 10 microns are the major cause of stickiness. Neumann's (24) analysis of the data of Andreasen, Hofmann-Bang and Rasmussen (1) and others seems to confirm this fact. One method of removing the adverse effect of these small particles is the addition of larger particles to the mass containing the fines. Hence stickiness is reduced either by dilution or by providing the small particles with a new surface upon which to adhere instead of flocking together. This scheme was tested by Hawksley (19) with a certain amount of success. Neumann (24) also showed that the flowability of fine powders could be improved by the addition of a uniformly sized adulterant.

In direct contrast, it has been found that the presence of a small amount of fines improves the flow rates of particulates. The data of Schwartzkopf (29) suggests that from 0 to 8 percent fines will give optimum flow rates depending upon the ratio of outlet diameter to particle diameter. Baeza (3) states "... that it is possible to reduce materially the excellent flow characteristics of a given powder by screening out of it a percentage of the fine or a percentage of the coarse particles . . ." This is supported by Hardy's measurements (18). It is interesting to note that the flow rates, just as the bulk densities, of particulates is constant for any number of different particle-size distributions (4, 18). However, improved flow rates have been obtained for particulates which approximate the distribution law of Andreasen and Andersen.

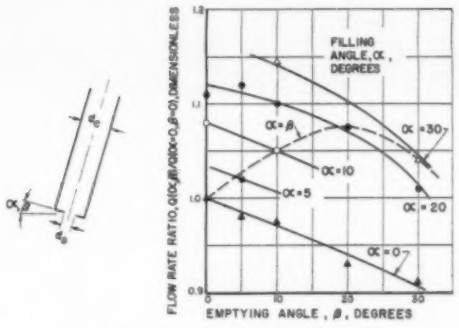
Wieghardt (31) observed that the flow of granular matter out of a vertical tube is slower than when the tube is slightly inclined. The flow out of the tube stopped when the angle of incline approaches the angle of repose of the particulate material, as expected. In Figure 6 are summarized the results of an extension of Wieghardt's observations. For the material studied, the optimum tube angle for filling and emptying was about 20 degrees. This optimum angle may well be a function of the angle of repose for the material flowing as well as the dimensions of the system. Certain other conclusions can also be deduced concerning the effect of the angles of filling and emptying on the flow rate.

Bingham and Wikoff (5) have found, for powders flowing through capillaries, that the flow rate increased as the capillary length increased; this effect becoming more pronounced as the particle size became smaller. It was thought that this was caused by the suction effect of the flowing particles.

In dealing with distributions of particles sizes, as

Figure 6

FLOW FROM NON-VERTICAL CHAMBERS



Material = sand
Bulk Density = 1.00 gm/ml
Void Fraction = 0.377
Particle Size = 590 to 840 microns
Static Angle of Repose = 35 degrees
d_c = 2.5 cm
d_o = 2.0 cm
Q = solids flow rate

compared to uniform particle sizes, there arises the problem of segregation with respect to the filling and emptying of containers. Zenz (35) has reviewed this problem with reference to Davies' (10) photographic studies. When a uniformly blended material is poured into a pile, segregation occurs with the fines essentially centrally located and the coarse material going to the sides. Should a bin, filled in this manner, be emptied, the initial discharge will be fines and the final discharge will be coarse material. Therefore the initial and final rates will be considerably different from that in between.

Finally, when hoppers containing granular material are emptied without disturbances, there many times remains a considerable amount of material adhered to the walls of the hopper. The use of disturbances to improve the flow out of the hopper can have adverse effects as well as beneficial ones if they are too drastic: erratic delivery and segregation being the more obvious possibilities. Brown and Hawksley (7) and Davies (10) found that flow uniformity could be greatly improved by the location of baffles and rods near the bottom of the hopper. The location of the baffles, their length and angle are very critical and depend upon the system with which one deals. Nevertheless, extremely uniform flow patterns are possible.

Conclusion

The major factors which affect the flow rate of particulates appear to be particle size, particle-size distribution, particle shape, density and surface characteristics as well as the relative size and geometry of the hopper. Flow rates can be improved by the correct choice of particle size with respect to the hopper dimensions and by controlling the particle-size distribution. Particle shape may not influence flow rates as much as might be expected (3). The most important single factor is the surface characteristics of the particles. Reduction of surface roughness and alleviation of stickiness are accomplished in different ways. Most common is the use of "lubricants" or "glidants", as they are sometimes called, which are added to the

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particulate mass. Such materials as graphite, talc, starch and waxes are often used. Their action may be to fill in the surface impressions of the particles, coat the surfaces or merely separate the particles from each other. In fact, the role played by the small percentage of fines, which have been found to improve the flow of particulates, may be that of filling in the surface impressions of the larger particles. A recently developed technique (33) for coating the surfaces of granules with smooth, dry surfaces can eliminate the necessity of adding "lubricants" to improve flow rates. The smoother the surfaces of the particles become, the better the flowability of the small size classes; hence improved flow rates through small openings can be obtained.

Although all reported investigations have dealt primarily with the emptying of containers, there appears to be no reason why the same factors which affect the flow of particulate materials out of a chamber should not similarly affect the flow into a chamber.

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The formulation of a sun-protective make-up base

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Today, the American woman has such a diversity of beauty embellishments and cosmetics that she is able to appear at her very best in all activities and under all conditions if she makes proper use of the appropriate preparations; and with the shortening of the work week and the many labor-saving devices for the home, both the career woman and the housewife have more free time for social and recreational activities. No matter how a woman spends her free time, she should do it by putting her "best face" forward.

Every woman is repeatedly exposed to the sun in her daily activities and especially in her vacation and recreational programs; and the full play of the sun's rays on the skin calls for great care in cosmetic resourcefulness. Not only must she use appropriate shades of make-up but she must also, in some way, protect her skin from excessive exposure to the sun that might result in a sunburn with subsequent redness, peeling, and irritation of her skin. On the beach it is common practice to use a protective cream or lotion containing a sunscreen. The majority of these products, while effective protective agents, do little to enhance one's appearance and are usually removed before "street make-up" is applied.

It was thought that if one of these same sun screens could be incorporated into regular make-up preparations, it might be possible for a woman to use such a dual purpose cosmetic to advantage. She could thus enjoy outdoor activities, wear her regular make-up, and still avoid the danger of sunburn. The potentialities of such a product would seem to be unlimited and public reception of a facial make-up with a "built in" sun screen is almost a certainty.

This project was undertaken with the hope of producing an acceptable make-up base with sunscreening properties.

A liquid make-up was considered the most desirable form and efforts were centered around this type of preparation. The basic problem was one of compatibilities, i.e.—combining the proper sunscreen agent with a base that would permit the incorporation of colored pigments to form a suspension of cosmetic acceptability. The ease of application to achieve proper make-up and the wearability of the make-up were criteria used in evaluating the final products.

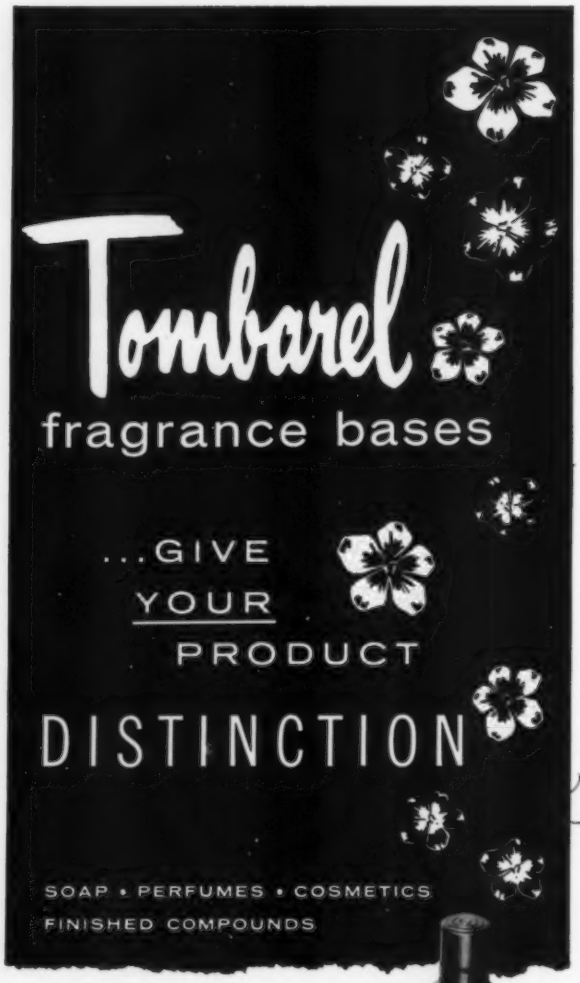
Originally, four basic formulations were studied: a simple stearic acid emulsion of the oil-in-water type, a perspiration-resistant make-up base previously developed in these laboratories[1], a lotion with a polyethylene glycol base, and a foundation lotion suggested by a supplier of cosmetic materials. The material chosen for use as a sunscreen was menthyl salicylate. A number of minor formula variations were made but none of the products was satisfactory in that they were either too greasy or they did not permit a smooth application to the face. Consequently, by trial and error, a new formula was developed and after many modifications an acceptable lotion type base resulted. The formula is listed below:

Lantrol	2.00
Polyethylene Glycol 4000	2.00
Stearic Acid	6.00
Cetyl Alcohol	1.00
Menthyl Salicylate	6.00
Triethanolamine	1.50
Methyl Paraben	0.20
Distilled Water	89.50

Method of Preparation:

All of the ingredients except the triethanolamine and water were mixed together and heated to a temperature of 75-80 degrees C. to effect complete

This paper is the result of a special project by Mrs. Torrico, a senior student in the School of Pharmacy at Purdue University.



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liquefaction. The water and emulsifier were heated separately to a temperature of 80 to 85 degrees C. and then poured slowly into the melted fats with steady stirring. Agitation was continued until a smooth cream was obtained.

This lotion, when applied to the face, leaves a thin film which serves both as a sunscreen and as a foundation for other make-up. Thus, this product offers two approaches to the problem of developing a sun screening make-up. If used without added pigments, the wearer can apply her own brand of make-up over it; or suitable colorants can be incorporated into this base to form a combination sunscreen and make-up in one product.

Compatibility and stability studies with cosmetic colorants were conducted and the products were found to be stable under normal conditions of use and storage. Since the experimenter was a blond with a fair complexion, a shade of make-up suitable for her personal use was developed. The following mixture* was used:

Cosmetic Russett C33-028	0.50
Cosmetic Tan C33-007	2.00
Cosmetic Sienna C33-023	1.00
Titanium Dioxide	33.00

It was found that a 10 per cent concentration of the mixture in the make-up base produced the desired shade.

It was found that the best way to add the pigment mixture to the make-up base was to make a paste of the colorant with glycerine and gradually add the lotion base to this mixture. Homogenization of the final product is necessary to insure complete uniformity of mixture.

Lantrol was found to be preferable to Lanolin in this formula because it gave a softer cream with less stickiness and no residual odor, yet appeared to aid in producing a mat effect. A higher weight polyethylene glycol was used in preference to the lower numbers because it gave better spreadability to the lotion. The stearic acid and cetyl alcohol were included as essential components of a foundation type lotion. Menthyl salicylate was the sunscreen of choice for two reasons: by virtue of its physical properties it was easily incorporated into the lotion, and it did not impart either a greasy feel or a gritty sensation when applied, as did several other agents originally considered.

Additional formulation studies may reveal other and better sunscreens than the one used in this particular formula; but this work has demonstrated the feasibility of combining sunscreens in facial make-up and it is to be expected that commercial products will soon appear on the market. A recent article in the *Pharmaceutical Journal*[2] reported the use of a "solar lipstick" containing sunscreens. Facial make-up of a similar type is the next logical step.

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* *These colorants are supplied by Ansbacher-Siegel & Co., Staten Island, New York.*

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Program for dermal toxicity testing

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RARE indeed is the client who comes to the New Drug Institute with a complete, well-designed program for conducting adequate and proper dermatological tests on his product. The reason for this is apparent. For the most part, management, medical directors, chemists, and even some pharmacologists representing industry lack knowledge about dermal testing procedures, their purposes and principles.

Many of these individuals are aware that methods for appraising the safety of dermatological preparations have been published, but they are vague as to how to go about applying these procedures to their particular product. Others complain that the procedures lack specificity and fail to furnish standards or yardsticks for measuring acceptance or rejection. They would prefer detailed specifications plus a set formula which, when applied to the data, establishes proof of safety without qualifications or qualms.

As a matter of fact, were a set of fixed and rigid procedures for the evaluation of safety to be published, instead of being useful and of considerable value to industry, it would in many cases lead to unfortunate and erroneous conclusions. For it must be recognized that no single pattern of tests serves to cover adequately the many ingenious chemicals and applications your industry markets to heal, improve, beautify, and adorn our skin and bodies.

I do not mean to suggest, however, that the design of each experiment and the testing of every new preparation be carried out without consideration of previous experience and published techniques. Uncharted seas are as dangerous to the pharmacologist as they are to a sailor. The point is, biological studies of any sort do not have the certainty of chemical and physical tests, their design is definitely and distinctly the subject of fine and careful selective judgment. Only the trained pharmacologist, possessed of years of experience in this particular field, is in a position to select the appropriate tests, or when it becomes necessary, improvise and design new tests suitable for evaluating the safety of a particular product. What criteria govern the pharmacologist confronted with the problem of laying out such a program?

First of all, in selecting a particular design of experiment, the investigator must be certain that the doses and other conditions of the test furnish data that will indicate the type of possible injury that may ensue, the extent or seriousness of the injury, and the margin of safety that prevails under the most extreme conditions of use. In short, he says to the test compound: "Do your worst" for only if he knows the potentialities of toxicity inherent in the preparation can he judge its relative safety.

Since humans are the ones who ultimately use these products, the ideal test subject for determining the safety of the product would, of course, be man himself. But man is not a guinea pig, and neither conscience nor the law allows his use as a test animal for initial toxicity studies. The danger that may unsuspectedly exist in untried products. the difficulties and high cost involved in conducting safe, controlled experiments with human volunteers make it imperative that we screen new materials and compounds by animal experimentation before risking human trials.

It must be recognized that the correlation between test results in man and in other animals is not always complete. Nevertheless, animal experimentation is our only resort and indeed is quite useful for relative comparisons. It permits us to predict, to some degree, the concentrations that may safely be tolerated by man. But there is a much more important and valuable dividend in animal experiments. In animals, the dose of the test material can be raised to the point of actual damage, and even death, something unthinkable in human trials. This *extreme* treatment makes it possible to discover, in a relatively short time, the type of injury that a smaller amount of the substance may only infrequently produce in man. In addition, toxic doses provide an estimate of the cosmetics, which may be employed by the young, the margin of safety under normal usage. Obviously,

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old, the fair, the dark, over large portions of the body for considerable periods of time, require a considerable margin of safety, more so than potent drugs. The latter are used for a comparatively short period of time under a physician's supervision. This is a factor commonly overlooked.

In the final analysis and on its most elementary basis, the *objective* of all pharmacological tests for dermal toxicity can be simply stated. All studies are directed at obtaining the answers to these questions:

1. Is there a likelihood of the test compound proving injurious to the user?
2. If so, to what degree and extent may injury be anticipated?
3. What form of injury does the damage take—sensitization, irritation, or systemic toxicity?
4. Is the injury reversible—does cessation of use, for example, reduce and alleviate the damage?
5. What consequences can ensue from special or unanticipated usage, such as accidental ingestion or if the preparation inadvertently enters the eye?
6. What is the margin of safety in event of excessive dosage?

A cosmetic may exert injurious action through its local effects or systemic effects, or both. Those effects that are local in extent are broadly termed irritations. They result from contact with substances which are primary irritants or sensitizing agents. A primary irritant is a substance which causes dermatitis by direct action on the skin at the site of contact. A skin sensitizer, on the other hand, is one that, without necessarily producing irritation on first contact, alters normal skin in such a way that application of the chemical on the same part or even other parts of the body produces a dermatitis.

A third type of local skin effect is "skin fatigue." The culpable compound does not produce primary irritation, but rather causes severe skin reactions after a number of exposures. Although it resembles sensitization, this agent is not considered a sensitizer, since, after a period of rest, the skin recovers its original resistance to injury.

The skin is an excellent barrier to the passage of chemicals but, in some instances, this barrier is breached or penetrated, and the compound enters the blood stream. Systemic dermal toxicity—where the body as a whole is affected—may occur following either single or multiple exposures to the test material. Whether or not a compound is capable of systemic toxicity is determined on the bases of signs and symptoms of poisoning, mortality, weight gain or loss, hematological studies, urinalysis, and gross and microscopic examination of vital organs and tissues. If systemic absorption is suspected, the propensity of the compound to cause body injury must be studied.

Tests most useful in evaluating the safety of dermatological products are described by Dr. John Draize of the Food and Drug Administration in his chapter on the subject in *Appraisal of the Safety of Chemicals In Foods, Drugs, and Cosmetics* (1). Dr. Draize presents a concise outline of the techniques now employed by the Division of Pharmacology. The current edition of this work is the third in a series. This indicates, more than any examples I can give, that procedures in dermal toxicity testing are not

rigid but are continuously being supplemented as new data are accumulated and evaluated. As further progress is made in the development or modification of existing techniques, new edditions, embodying these advances, can be anticipated.

Dermal toxicity testing

To elucidate our present thinking on the subject—dermal toxicity tests can be divided into a series of categories, each designed, broadly speaking, to determine a particular avenue of potential toxicity. The order in which the tests are performed is based on economic considerations. Thus, the simpler and more inexpensive tests are first recommended; these must first prove favorable before more complex and more extensive tests are undertaken. The results are cumulative; they serve as bricks with which we construct an edifice of safety data.

To burden you with the technical details of these tests would be boring to those of you who are familiar with the techniques and scarcely helpful to those whose knowledge may not extend to these particular operations. Yet a brief outline of the general scheme of dermal toxicity testing may well serve as a refresher to many and a guide to those who may have to concern themselves, in the future, with various aspects of the problems involved. I should, however, like to elaborate upon several newer tests that we have developed or employed in our laboratories.

Animal primary irritation tests fall into several categories. The skin of rabbits responds similarly to man's. This suggests their use for patch testing, using both abraded and intact skin. More delicate than the patch test is the trypan blue test (2, 3). By means of this technique, differences between substances which cannot be discerned by the patch test readily become apparent. This dye, introduced intravenously, seeks out and stains even mildly irritated areas because of their increased vascular permeability.

Where an evaluation of primary irritation of mucous membranes is sought, the tissue of choice is the specific mucosa to which the product is to be finally applied. Differences in histology, absorptive capacity, secretions and pH ranges makes the transferance of data extremely hazardous. For oral mucosa, the test material is applied to the buccal surface of rabbits. Vaginal preparations are tested in dogs, penile and rectal preparations in rabbits, and eye preparations in rabbits.

Eye irritation studies are necessary for all preparations which may be used in or about, or inadvertenty get into, the eyes.

It should be emphasized, however, that none of these irritation tests, which consider erythema and edema, provide adequate data concerning the transient stinging or smarting sometimes observed when chemicals are applied to mucous membranes or abraded skin. These unpleasant sensations can occur at concentrations well below levels that produce objective evidence of local tissue injury.

An eye closure test, using rabbits, has been developed in our laboratory to analyze the pain parameter of chemical irritants applied topically. In this procedure, the unknown material is placed in the conjunctival sac of a rabbit's eye. A positive reaction is scored when the rabbit keeps its eye closed for a fixed period of time following the administration of the drug.

A reaction which is of growing concern to the pharmacologist is sensitization. The potential capacity of a new compound to sensitize the user can be readily screened in a guinea pig test. By this technique, moderate to strong sensitizing agents, apt to affect a large proportion of the population, can readily be distinguished from mild sensitizers.

Usually reserved for preparations containing high concentrations of extremely active components is the single dose dermal toxicity test in rabbits. Results are reported in terms of LD_{50}. Also, where *absorption* is suspected, oral feeding in rats is useful, at least for the light it throws on the potential systemic toxicity of the compound.

Since cosmetic materials are intended for more or less continuous use, consideration must be given to long-term toxicity studies.

The first of these tests is the 20-day dermal toxicity study. Its purpose is to evaluate both the local and systemic toxicity of products used infrequently or under medical supervision. The test material is applied daily for three weeks to intact and abraded skin of rabbits over approximately 10% of the body surface. Body weight, food consumption, condition of the skin, and signs and symptoms of poisoning are reported. Urinalysis and hematological examinations are conducted both before and after treatment. Gross and microscopic examination of vital organs are conducted on the animals who die or are sacrificed at the end of the experiment.

A more rigorous test is the 90-day dermal toxicity test. This is designed to evaluate dermatological preparations intended for application to the skin for prolonged periods of time. Skin ointments and cosmetics may fall within this group. Application of the test material is made daily for 90 days to the intact skin of rabbits. The long period of treatment is designed to detect and evaluate agents which may produce progressive deterioration. The same observations and tests on the subjects are made as in the 20-day dermal toxicity test.

Substantial savings in time and money can be made by conducting the 20 and 90-day study as a single combined experiment. A sufficient number of animals and levels of treatment are started so that only a portion of the animals need be sacrificed after 20 days. The rest of the animals are continued on the treatment for an additional 70 days.

There are, of course, some risks in this combined study. The preparation may be considerably more toxic than anticipated with the result that the doses selected for the 90-day test do not yield adequate information about the maximum safe dosage. Fortunately, unexpected toxicity does not occur often. If it does, the product is probably unsuitable.

This brief review of the procedures available for testing dermal toxicity is not, of course, the full story. The data obtained usually are without significance to the uninformed. These experiments must be considered incomplete unless an experienced pharmacologist interprets the results. Only his judgment can

be relied upon in deciding whether human trials are advisable.

Let us see how the trained pharmacologist goes about reaching his conclusions. Testing the toxicity of thioglycolates, Dr. Draize and his co-workers (4) found that death (preceded by severe depression) occurred between the third and tenth days in 11 of the 18 animals receiving only twice the equivalent of the "human dose." In fact, at the "human dose" level, 2 of 17 animals died within four weeks. All survivors sustained minor local and systemic effects.

At first glance, these results seem alarming, and the material quite toxic, yet Dr. Draize concluded that "except in the case of sensitive individuals, the judicious use of these solutions should prove a relatively innocuous procedure."

What criteria did Dr. Draize employ? Undoubtedly, he took into account, (1) the chemical and physical properties of the material, (2) its method of administration and application, (3) its systemic dermal toxicity, (4) the expected frequency and duration of exposure to the material, and (5) the condition of the skin at the time the material was to be applied; that is, would the product be used on normal skin or on skin which may be irritated due to sun, wind or pathological conditions. After weighing each of these factors, he decided that the animals were poisoned under conditions considerably more rigorous than those anticipated in the product's use. His conclusion followed as a matter of discerning judgment.

With regard to primary irritation and sensitization tests in humans, there are several schools of thought regarding procedures to be used in conducting the sensitization portion of the studies. Dr. Draize recommends that the dosage schedule for a sensitization study in man follow the procedures usually employed for the guinea pig sensitization test. In the case of man, however, the test compound is applied under a patch. The information obtained upon reading the skin reaction after the first patch is used for evaluating the primary irritation of the compound with respect to man.

To assure that the findings of the sensitization test be statistically significant, Dr. Draize recommends that at least 200 individuals, evenly divided as to sex and encompassing as wide an age range as possible be employed. Under these circumstances, very weak sensitizing agents can be readily recognized.

To avoid the inconvenience and expenses involved in requiring 200 subjects to undergo a series of 11 patch applications, many dermatologists employ a modification of the Schwartz Prophetic Patch Test (5). One modification requires the initial patch be kept in place for 48 hours. The reaction at the site is noted and observed again after 48 hours. Two weeks after the first patch has been applied, the test is repeated on the same subjects and on approximately the same skin area. The patch is again removed after 48 hours, and the sites examined for signs of increased sensitivity over a period of an additional 48 hours.

Although this technique is not as stringent as a repeated patch test technique, it is useful for detecting mild to moderate sensitizing agents.

Other dermatologists use the repeated patch testing technique but only employ 25 or 50 subjects. Even with the smaller number of subjects, mild and moderate sensitizing agents can be discovered.

Aside from the ability of the repeated patch test technique to uncover agents responsible for skin fatigue, there seems to be little *difference* in terms of information gained or of cost between the repeated patch test employing, say, 36 subjects, and the Prophetic Patch Test, using 200 subjects. The number of patches applied is approximately the same.

In any case, the question of senitization is fundamentally a relative matter. The incidence of positive responses to a particular agent can be increased or decreased depending on (1) whether or not the patches are reapplied to the same site, (2) applied as closed or open patches (in a closed patch, the test material is sealed to the skin on all sides by inserting a thin sheet of rubber or plastic between the patch and the covering bandage), (3) the length of time the patches are left in place, (4) the condition of the skin at the site of application of the patch, (5) the presence or absence of other irritating agents under the patch, (6) the concentration or amount of material placed under the patch, and (7) the number of repeated patch insults.

Thus, it would appear that the ability of the dermatologist conducting the test and making the evaluation is more important than the particular technique employed.

Many so-called "human use tests" are neither convincing nor scientific so far as judging the safety of a compound. To rely exclusively on whether or not few or no consumer complaints are received following distribution of the sample in a test market, is wholly unjustified. Nor are the number of units sold over a short period of time an indication of safety or acceptability. Rather, this information is often merely a tribute to the potential usefulness and successful promotion of the product. Most people, after experiencing a reaction to a product, will simply fail to make a second purchase. Few report their dissatisfaction to the firm unless the reaction is sufficient to warrant medical attention. Only when the consumer is induced to reply to a questionnaire so that an estimate of the incidence and severity of reactions can be made, can the "use test" have validity.

I can only conclude this brief discussion of dermal toxicity by emphasizing once again that an experimental study is merely a collection of data and is incomplete until the results have been evaluated and interpreted by an experienced investigator. There is no simple mathematical formula to judge toxicity. The most important part of the study is truly the integrity, training, experience, competency and judgment of the pharmacologist.

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Abstracts from the

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The psychology of odour and successful perfuming.

Dr. E. Paukner, Munchen

Much has been written about the relation between the phenomenon of scent and the human psyche. Rarely has an attempt been made to establish basic principles. The theories which have so far been put forward on this difficult and complex subject of odour psychology are hardly likely to encourage further research on it. However, market research is continually trying to gain an insight into the mental associations and imagery aroused in the consumer by the scent of a certain product. Up to now, the market research worker has had to use empirical methods of doubtful value for odour testing; for research on fundamental principles a practicable terminology of scent must first be established, for expert's terminology cannot be used with the public. Since there are no physicochemical methods by which odours can be classified, language must provide criteria of quality and of the effect of scent on the human being. Should this succeed, a classification of odours and the establishment of terms, which would awake in all human beings the same response and image, should be possible. The difficulty of such an investigation lies in the first place in obtaining a representative panel, large enough to render statistical interpretation significant. The questions posed in carrying out the work were: Are there any scent characteristics, to which a panel reacted in a similar or even identical way? Do such odour characteristics produce identical descriptions? A test sheet with 66 adjectives was prepared in order to provide comparable terms, instead of allowing free description and association. The test panel were asked to classify the six test odours under the given headings, according to the character of the odour and their reaction to it. The six odours chosen were clearly defined chemically and thus reproduceable odours were guaranteed. They represented the following odour types: citric, floral, spicy, minty, animal and green. The interpretation of the experiments aimed at evaluating the results of the panel, rather than individual observations. Men and women were tested separately by means of various statistical methods. It was shown that blank tests carried out with coloured and uncoloured water gave a certain picture, which differed substantially from the results achieved with scent. The results of the allocation of adjectives to each scent were presented in profile form and their significance discussed. Finally some terms were established, which appeared to be typical for each individual scent. Since it is also important for market research to have information about the connection between scent and colour, the tests were enlarged to include colour. The results corresponded to a large extent.

The method arrived at has made it possible to establish specific effects of odour, at least in the case of uniform compounds. It has led from empirical to scientific nomenclature by making use of language, the widest approach there is to the human psyche. As part of cosmetology, odour psychology must have fundamental principles; this paper is a contribution in this field and after further expansions promises interesting results.

In the course of the lecture, the scents used in the tests will be demonstrated to the audience by spraying.

Skin and psyche.

Priv.-Doz. Dr. Dr. S. Borelli, Munchen

When this topic is discussed, either the action of emotional factors on the skin or else that of especially chronic skin diseases on the emotional condition may be envisaged. It is difficult to rationally determine psychogenic effects. In case of prolonged diseases action and reaction will take the form of a vicious circle. The factor which released the initial symptom is rarely found. Emotional changes due to chronic skin diseases may result from a feeling of personal impairment and inferiority or from the actual fact of offending.

There are numerous publications concerning the

action of emotional factors on the skin, whereas the number of papers related to the effect of skin conditions on emotional stability is comparatively reduced. It will be tried to give a summary of the existing literature to this effect in combination with the author's own experience.

Contribution to the analysis of creams containing various stearates.

Dr. Pierre Velon and Chem.-Ing. Jacqueline Picot, Paris

The authors deal with a method worked out by J. E. Wolff, for the analysis of creams containing stearin, alkaline stearates and triethanolamine stearate.

This method by Wolff consists of two determinations:

1. The determination of total fatty acids and triethanolamine, by addition of alcoholic KOH and backtitrating with HC1 and
2. the estimation of total fatty acids by acid hydrolysis carried out by boiling. It was investigated, whether this method could be used if the cream contained a stearic acid ester i.e. glycolstearate. This was tested by titrating the hydrolysed glycolmonostearate in a) aqueous and b) aqueous-alcoholic medium.

It appears that Wolff's method makes it possible to titrate free stearin, sodium- and potassium stearate, triethanolaminestearate, and glycolstearate in creams. In practice it has, however, been found that after acid hydrolysis of the cream, the fatty acid values are 10% below the theoretical value. It would appear that the glycolstearate hydrolysis, in the presence of concentrated HC1 and an excess of stearate, is incomplete and that one is confronted by the well known equilibrium between esterification and hydrolysis.

It has now been attempted to work in an alkaline rather than an acid medium. Two methods can be used:

The first method consists of estimating the free fatty acid and that linked to the triethanolamine, by backtitrating the cream which has been dissolved in excess alcoholic KOH and boiled for one hour; then estimating the total fatty acids by liberating the fatty acids by hydrolysis from the residue which has been dissolved in alcoholic KOH and backtitrating with HC1. This still leaves the stearic acids of the glycolstearates to be estimated. The free stearic acids and triethanolaminstearates are converted to alkalisoaps, which are decomposed by HC1, whereas the glycolstearate remains unchanged.

The second method is more sensitive and allows the determination of the triethanolamine in the washings. In the first method this is only possible by calculating the difference. The estimation of the fatty acids and the triethanolamine correspond to the first method. Total fatty acids are determined by precipitating the fatty acids, dissolving them and titrating. The estimation of triethanolamine and the fatty acids, apart from those combined with glycol is identical with that given in the first method. The triethanolamine, present in the washings as chlorhydrate, is titrated after the fatty acids have been filtered off.

Each step in this method is predetermined by an equation.

Thus, when analysing unknown creams, the analysis can be totalled up and compared and it can be seen if the cream contains saponifiable products or not. Results are quoted for a cream with glycolstearate, but the method is also applicable in the presence of vegetable oils or partially or completely saponifiable products.

Both method 1 and 2 can be used; the second method has however the advantage of offering direct determination of triethanolamine, which is often only present in the cream in very small amounts.

Measurement of the water-index of lanolines and vaselines containing cholesterin.

Dr. Pierre Velon and Chem.-Ing. Jacqueline Picot, Paris

To measure the water-index of lanolines and vaselines containing cholesterol the "ball-test" method has been developed. In the test of 1954 water had been added in surplus, the non-absorbed water was hurled off and the emulsion was weighed. The difference showed the absorbed water.

This method is difficult because it is not possible to absorb the surplus water with filter paper in vaseline. Vaseline would stick to the paper. The authors simplified their method using now an index which characterizes the end of the reaction. Important in this test is the species of the vessel (stainless steel) and of the spatula (horn).

Measuring: Addition of H_2O dest. (½ ml at any

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time) and stirring steadily and energetically. At first the water will be absorbed quickly, later on it slows down. The end of the test is given by that moment, when the water will not be absorbed during 3-4 min., emulsion glides on the sides of the vessel and adstrings round the spatula in the form of a ball. Volume of the consumed water shows the burette. (Water-index = volume of water which can be absorbed by 100 g of an absorption-base). During the measuring the temperature shall be constant (20° Celsius).

This method can be used for lanolines and other absorption-bases and for vaselines containing cholesterol.

Comparable results need a standard-vaseline for different cholesterols or a standard-cholesterol for different vaselines. (0,1 g cholesterol—10 g vaseline, heated on 150° Celsius and cooled to 20° Celsius). The exactness of this determination for vaselines and for vaselines containing cholesterol is ±2%.

The use of ternary Diagrams in cosmetic formulation.

R. James and R. Goldemberg, USA

The efficiency of a research program depends on the extent to which all work done is purposeful and leads to the desired goal with a minimum of random effort. This paper offers a logical approach towards fulfilling these requirements through the use of ternary diagrams combined with the concept of "parallel" and "crossed" series of experiments.

The basis of the approach is the fact, that each new series of experiments may be related to previous ones in two ways: they may be paralleled or they may be crossed. During the initial stages of an investigation the use of "crossed series" of experiments will quickly define the area in which the most desirable formulas may be found. When this area has been defined, a single set of "paralleled series" will usually spot the single most desirable formula.

Combining this concept of crossed and paralleled series of experiments with the use of ternary diagrams to record data reduced the number of samples required to obtain desired information. This is true because the ternary diagram can demonstrate three variables simultaneously in a way that a fourth variable can usually be estimated. The result is that information is condensed into less space, affording the formulator a clearer understanding of the system under investigation.

The influence of HLB-System on the activity of ointments.

Prof. Dr. H. v. Czetsch Lindenwald, Alexandria

Our new ointment-experiments are demonstrating again that ointments are not simple solutions or suspensions but colloids. The type of colloid is of capital influence on the added materials. A criterion of the desired properties of an ointment or cream is given by the HLB-System.

By this way it is possible to predetermine with probability spreading, the character of emulsions and beyond it the resistance against abrasion and washing.

For all ointments it is highly necessary to take care for adsorptions. They also depend on HLB-System. Hydrophyle ointment-gels particularly often are reacting in another manner than solutions, so that new aspects must be respected absolutely by combining preparations including active materials.

IR-spectrometric analyse of oleaginous substances and bases for cosmetic preparations.

Prof. Dr. F. Neuwald and K. Maennchen, Hamburg

IR-spectra of a series of natural and hardened as halfsynthetic fats and oils, of ointment bases, and of nonionogenic emulsifying agents on base of esters of fatty acids are reproduced and discussed.

The use of cosmetics in preventive medicine.

Dozent Dipl. Ing. L. Th. Schonagen, Leiden

An ample survey is given, explanated with some examples, about the preventive medicine in general and the aim and utility of the preventive medicine in particular.

The cosmetology has an important function in the preventive medicine, but this is also the matter turned up. Will a cosmetic preparation satisfy the expectations, than at first it must be tested preventive medicinal about toxicological reactions, the cause of allergic reactions and other phenomenons of not-suffer or over-sensibility. In particular it must be tested, if it is tenable, so that the preventive medicinal investigation had to be repeated in several periods during the time after the preparation. At the same time it is necessary to search the exact directions for use of a fixed preparation to prevent phenomenons of sensibility, that could appear by a strange method of use.

The use of cosmetics in preventive medicine is very important, not only in the private section but also in the working-psychology and the occupational physiology. Namely it advances one of the most important things in preventive medicine "the general sanitation", but they can also prevent bodily defects for instance of the skin, the hair and the teeth, by proper choice and use. The psychological importance of the use of cosmetics is extraordinarily great. Nervous and physical tensions cannot only be prevented, but also the healing of the individual can be increased, stimulated and promoted, specially of women. (The healing, however, does not belong to the activity of preventive medicine.)

In the Netherlands an ample research was made of the women skin. The collected information was statistically worked and from it conclusions were taken with respect to the demands of preventive working, that in the Netherlands must be prescribed as skin-cosmetics. The alternating standard of the N.M.F. in the skin seemed to be of great importance, so that it is necessary that the cosmetics for skinfood must be so prepared that they have a corrective working on the alternating standard of the N.M.F. in the skin, to give the security of a good preventive medicinal effect.

The statistical research proved that geographical and topographical differences have an important influence for instance the temperature and the composition of the air (specially in the environs of the industry). With good reason can be concluded, the

use of cosmetics in preventive medicine can give important results.

Animal experimental investigations upon the influence of certain biologically active substances on the metabolism.

Dr. H. Eder, Frankfurt/Main

For the development of new cosmetic preparations it is of some importance to elucidate the effective mechanism of their biologically active substances. In an increasing degree objective measuring methods are employed, in order to investigate the influence of the biologically active substances on certain physiological processes of the human (or animal) organism. The obtained results can then be evaluated as supplement of the results of clinical tests or can show whether or not clinical tests would be recommendable.

For the investigation upon the effect of certain biologically active substances the indirect animal calorimetry is particularly suitable.

By means of measuring the gaseous metabolism, the energy transformation of the animal and the metabolism of the animal and the metabolism of the single nutritive substances can be determined. An influence of the metabolism by biologically active substances is recorded by a systematic variation of the physiological values.

For an exact registration of these data, an apparatus has been developed which is suitable for small laboratory animals. This apparatus allows the performance of metabolism investigations of physiological, pharmacological and toxicological nature with a high degree of exactness under observation of optimal conditions.

In a closed system, the CO_2 concentration of the circulated air is first measured continuously by means of an infrared absorption recorder and the CO_2 then quantitatively absorbed. From the throughput of air and the CO_2 concentration the CO_2 discharge of the animal can be determined. The oxygen is fed into the closed system in measurable quantities. It is generated in an electrolytic cell in amounts just compensating the oxygen requirements of the animal. The electrolytic current is measured and recorded. It is rigorously proportional to the amount of oxygen generated and thus provides an exact measure of oxygen consumed by the animal.

The concentration of the CO_2, of the electrolytic current, as well as the motoric activity of the animal and the temperature of the environment are registered. In this way curves are obtained which can be graphically analyzed by a planimeter for any desired time intervals. In addition, amperehour meters sum up the electrical output and allow a direct reading of the values of the gaseous metabolism for defined periods of time.

The measuring principle and the equipment provide conditions that are similar to natural ones. The measuring error of the CO_2 measurement is 1.1 to 1.6 per cent at maximum of the individual values recorded; in the oxygen measurements it is merely a function of the recording accuracy of the measuring instrument.

After a period of preparation of one week, during which the animals are put on a test diet and kept in the same room in separate glasses at 28° Celsius, they are put before the proper experiment into the animal containers for two days where they are made familiar with the test conditions. Normally, the test temperature is 28° Celsius. Each measurement takes 24 hours. Measurements effected within shorter periods are bound to lead to errors due to the diurnal rhythm or spontaneous fluctuations in the gaseous metabolism. For an accurate indirect calorimetry it is necessary to determine the amount of nitrogen discharged in the 24-h-urine in addition to the values of the gaseous metabolism during 24 hours. The recorded CO_2 values are standardized by conversion into standard values cmm/qcm surface/min. The energy-transformation is calculated from the caloric oxygen values on the basis of the corrected respiratory quotients resulting when the protein metabolism is taken into account.

We have also investigated the effects of royal jelly by means of the described apparatus, due to the numerous reports on the multiple cosmetical and clinical usability of this substance.

Consumer research on new products.

Dr. R. H. Marriott, London.

Testing products by a consumer panel was well reviewed at the British Congress of Cosmetic Science by L. H. Ovens. It is reasonable to suggest that such procedures of evaluating products by simple consumer reactions are not altogether complete methods. In fact, experience indicates that such testing by miniature panels is more likely to say "no" than to say "yes". Many best-selling lines—and this is the only true measure of acceptance—would never have been launched if panel testing had been in operation.

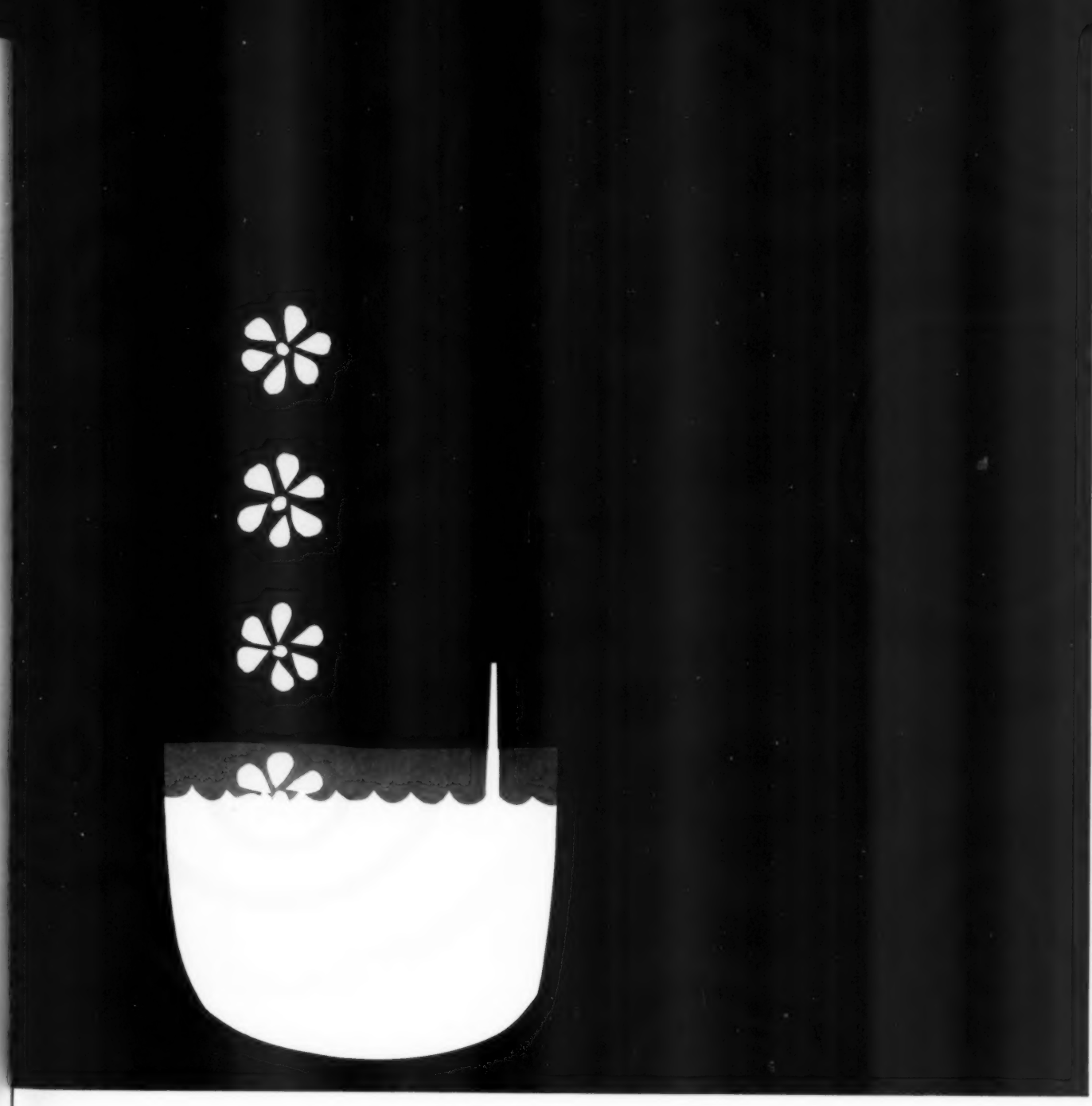
That doubts are felt on the general modus operandi is shown by Powers' paper in the same Congress. Here he indicates that there is more to it than ordinary consumer research technique in that he advocates a positive utilization of laboratory testing to clarify the goodness of the proposed product.

In the same way there is much to be said for introducing into the minds of the panel members the features for which some positive claim may be made and to find out whether the user can or cannot agree with the justice of the claim. Without this background it is clear that advertising becomes guesswork and may even lead to the death of the product.

The present trend of "follow my leader" approach in the marketing of cosmetics, ably assisted by the consumer research methods of comparing one against another inevitably leads to a high standard of mediocrity with a concomitant dearth of outstanding innovations. It may lead towards the introduction of "wonder" ingredients but these are all of a kind and to expect an uninformed panel to be able, by observation, to appreciate an undefined something is an act of credulity beyond reason.

It is suggested that products which are to be panel tested have to be considered in the light of whether

a) they are to be replacements of an existing product,



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b) they are to be improvements of an existing product,
c) they are to be novel and fulfil requirements which hitherto have not been envisaged.

Those in class a) are probably best looked after by the standard tests. Those in class b) by a modification of standard testing. New and novel products cannot genuinely be compared with existing ones and in the testing they have to stand on their own ground but tested by a panel of users who have been informed.

Spray patterns of cosmetic pressure packs.

A. Herzka, B. Sc., A.R.I.C., London.

This lecture deals with spray pattern and summarizes the ways and means to achieve correct results.

The more important factors which control the type of spray obtained are:

1. the nature of the propellant, i.e., whether a liquefied or compressed gas, or a mixture of both types,
2. the ratio of propellant to product,
3. the headspace pressure,
4. the valve design, and
5. the viscosity of the product.

Some of these factors are more important than others when combined differently in order to achieve a definite purpose. Thus when employing a liquefied gas propellant, the headspace pressure is not as important as on those occasions when a compressed gas is employed. But even with a compressed gas, the headspace pressure is nowhere near as important with liquids of low viscosity as with those that are highly viscous.

In order to illustrate these points the main types of pack that are likely to be encountered in the cosmetic and allied industries are dealt with.

For **surface sprays,** standard valves either with ordinary actuators, or with break-up spray actuators are employed in order to give the appropriate spray pattern, and the choice of the actuator **is** governed by the quantity and type of propellant. An alteration in the type of actuator employed not only affects the spray pattern in both planes, but also has so called side effects which are equally important, namely an alteration in the particle size and in the flammability rating.

For **foams,** standard valves with foam actuators are employed. These foam actuators are characterized by large internal expansion chambers which continue without interruption into the large external orifices. Products packed with foam actuators will yield a foam immediately after emission from the external orifices. Alternatively, a foam on impingement can be achieved by using a spray actuator rather than the foam actuator. That type is more common with glass packs, as very few foam actuators are suitable for use with 20 mm valve closures.

For **streams,** the special actuators developed principally for nitrogen packs must be employed and in order to achieve correct formulation it is necessary to keep a close check on the viscosity of the product and to determine very carefully the pressure that is necessary to expel most of the product residues, i.e. the

SPEAKING FROM SPAIN

Carlos Susanna Gregory is a graduate of the University of Barcelona. Since 1951 he has been employed with cosmetic firms and is now manager of production with Synox Laboratorium. He is present Secretary of the Society of Cosmetic Chemists of Spain.

Glycerine versatility

IN DEODORANTS... ANTIPERSPIRANTS... MOUTH WASHES EYE LOTIONS...TOILET WATERS... NAIL LACQUER REMOVERS

The extensive range of useful glycerine properties makes it a desirable ingredient for the cosmetic chemist and the pharmacist in many formulations. Its function may depend on the chemical and physical characteristics of the vehicle, or on the softening emollient action it is capable of exercising on the cosmetic film brought into contact with the skin.

Many papers have been published on the cosmetic applications of glycerine[1, 2, 3, 4, 5, 6, 7, 8] the detailed description of which we will omit here. In this article we will attempt to cover only the important and versatile role which glycerine can play in formulations of deodorants–antiperspirants, mouth washes, eye lotions, toilet waters and nail lacquer removers, following mainly the European criterion.

In order to clarify the subject somewhat, we have outlined a formulation scheme on the products to be reviewed, with their usual forms of use, the chief characteristics required, and the typical ingredients (see table, pages 2-3).

DEODORANTS – ANTIPERSPIRANTS

Within this line of products, three different presentation forms are popular, in each of which we will examine the functions and advantages of glycerine.

a) Sticks: The high alcohol content of these solidified products, of the type of an alcohol-stearate gel, presents the problem of controlling its easy volatility. This can be achieved in some degree by addition of glycerine, a polyalcohol with very low volatility. Such an addition of glycerine also has a plasticizing action which protects the stick from breakage, and, as a non-volatile solvent, glycerine prevents the settling of sodium stearate to the skin.[9] Another effect, also due to the addition of glycerine, is the maintenance of an adequate degree of transparency of the stick.[10] The moistening effect of glycerine prevents the too rapid drying of the deodorant on the skin, giving to the stick a smooth-touch and favoring its application.[8, 11] It is normally advisable to employ no more than 5% of glycerine, since it may excessively retard the necessary evaporation rate in use, reducing thereby the refreshing effect of the product.

The function of glycerine in controlling the evaporation also prevents the possible crystalization of the astringent salt, which if it were to happen, would create difficult application problems.

Another interesting effect is that glycerine can act as solvent for the gelation agent (or solidification agent), permitting increased concentration. This makes possible an elevation of the melting point of the stick, an important consideration in the warm countries.

b) Creams and Lotions (solid and liquid emulsions): The so-called vanishing creams constitute the type with most widespread acceptance (O/W emulsions). The frequent inclusion of astringent salts and bactericides into such products creates some additional problems, which must be taken into account when formulating said products: in the first place the presence of astringent salts, in many instances characterized by a strong acid hydrolysis, prohibits the use of usual emulsifiers of the soap type (TEA stearate, potassium stearate, etc.) and makes necessary the use of emulsifiers stable to acids (for instance: sorbitan esters and polyoxyethylene sorbitan esters, the acid-stable glyceryl mono-stearate, some anionics, etc.). In the second case, the presence of germicidal agents (especially the phenolic types) produces inactivation phenomena in the presence of nonionic emulsifiers.[12, 13, 14, 15, 16, 17]

In spite of inclusion of astringent salts and germicidal agents, these creams continue to exhibit the usual characteristics and difficulties of the vanishing creams: ease of water evaporation from the external phase of emulsion and consequent drying, consistency alterations, possible shrinkage of product from the walls of the container and sometimes appearance of crusts on the surface of product. These difficulties must be overcome with a moisturizing agent such as glycerine. To quote deNavarre,[21] introduction of glycerine in O/W emulsions gives a better fluidity (especially in liquid emulsions) and plasticity than addition of other humectants, so that glycerine seems to favor formation of O/W emulsions, contrary to propylene glycol, which seems to favor formation of W/O emulsions.[22] As to the influence of glycerine in counteracting the hardening of an O/W emulsion, it has been found[23] that the optimum concentrations are 7.5% for glycerine, 15% for sorbitol and 10-12% for propylene glycol, respectively. About the question of influence of the diverse polyols on viscosity of an emulsion, the opinions are divergent.[2, 18, 21, 23, 24, 25] In our own tests we have found that association and blending of glycerine with equivalent quantities of polyethylene glycol 300 or 400 still improved the plasticizing properties and spreading of a vanishing cream.

Again, the acceptance of glycerine as humectant is shown by the fact, that Wells[8] maintains even that it can act as emulsifying adjutant and improve the stability (especially in O/W emulsions with polyoxyethylene alkyl ethers). It has been mentioned as another function of glycerine, that, if the emulsion breaks or tends to invert, the glycerine favors the phase inversion, maintaining homogeneous and plastic the resulting new film.

A further effect of its moisturizing action,[26] and not the least in importance, is its softening emollient action on the dry skin, extensively proved by the well known work of Blank.[27] Moreover, glycerine has the subsidiary action of preventing the "rolling" due to the poor adhesion of the film to the skin. Pepper[28] also mentions the value of incorporating it into the vanishing creams assigned to cold countries, since it prevents the breaking of the emulsion, thanks to its ability to lower freezing points.

Product	Principal use	Required characteristics
Deodorants-Antiperspirants	Mask or destroy the secretion odour, or prevent it.	Good consistency, ease of spreading, and such form of package that prevents evaporation. Must not grease or soil clothes.
		Ease of spreading, in addition to its specific properties.
Mouth Washes	Oral hygiene, cleaning, refreshing and/or odour masking action.	Ease of use and efficacy.
Eye Lotions	Clean, stimulate, refresh, soften the membranes of the eyes.	Must not irritate, must be stable, neutral pH, isotonic with lachrymal secretion, and clearness.
Toilet Waters and Stick Colognes	Perfume the body.	Ease of use and pleasant appearance.
Nail Lacquer Removers	Dissolve lacquers.	Careful package preventing evaporation. Must not dry the nails too much.

It can be mentioned too[29] that glycerine has been recommended as a solvent for the new bactericide based deodorants (chlorinated phenol derivatives).

As seems apparent from all this, glycerine supplies in a balanced manner all the properties (hygroscopicity, humectant range, viscosity, low viscosity index, good compatibility, low volatility, lack of toxicity, good colour, taste and odour, lack of corrosive action, low freezing point, required by Griffin[18] of ideal humectant agents.

We believe that the selection of glycerine as moistening agent for deodorant-antiperspirant creams and lotions has so been justified.

c) Liquids: Such aqueous or aqueous-alcoholic solutions require addition of glycerine for its moisturizing, emollient action. Its spreading action favours good adhesion and wetting of the skin. Furthermore, when it is included in sufficient quantity, it is able to impart an adequate body to the lotion or to enhance the effects of other thickening agents.

MOUTH WASHES

Inclusion of glycerine in such preparations usually has various missions: it increases vehicle viscosity, exercises a moisturizing action, acts as solvent and diluent for flavoring materials, and gives a sweet flavor permitting sometimes a lower concentration of other sweetening agents. Wells[8] mentions also its utility as "throat conditioner," and its use in lozenges for this purpose is well established.

EYE LOTIONS

Often these lotions simply consist of a physiological sal solution; in other cases 0.5% methyl cellulose solutions are used, which give an inert, viscous colloid of good tolerance not causing allergic reactions. Addition of up to 10% glycerine can be of interest for its isotonic effect and spreading action. It is a constituent of proprietary products for such use.

TOILET WATERS

Introduction of a low percentage of glycerine into toilet waters increases or maintains solubility especially when the product is subject to evaporation.

Another reason for use of glycerine is to retard perfume volatilization, to fix it somewhat, thanks to its solvent properties and low vapour pressure. Moreover, it must be borne in mind that, due to its compatibility, it does not produce any alteration of perfume.

NAIL LACQUER REMOVER

Addition of glycerine to these preparations is indicated by its emollient action, imparting unctuosity without oily character, a very important feature. This lubricating effect partially compensates for the unpleasant dryness of the skin produced by strong solvents, the basis of most of these products.

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Vehicle	Typical components		
Stick, solidified alcohol gel.	solidifying agent 5-10% glycerine 0-5% alcohol, perfume qs.	astringent salts: Al-salts 15-20 (sodium stearate) germicides: G-11 (0.25%), anobial (0.5%), Chlorophyll (0.05%), quaternary ammonium (2.0%)	
Cream O/W vanishing acid type. Also lotions, liquid emulsions.	Oil phase 15-20% typical ingredients germicides	Aqueous phase 85-80% Glycerine 5-15% astringent 15-20% Fabric-damage inhibitor 5-10%	Emulsifier acid-stable nonionic (incompat.)
Liquid, aqueous or aqueous-alcoholic solutions.	germicide agent astringent agent 15-20% glycerine 3-15%		
Aqueous or aqueous alcoholic solutions. Concentrated liquids.	antiseptic and astringent substances glycerine 0-5% flavoring material		
Buffered and isotonic aqueous solutions.	weak antiseptic, astringent agents, vasoconstrictors glycerine 0-10%		
Liquids.	alcohol, perfume, water q.s. glycerine 0-5%		
Stick.	(as for deodorant sticks)		
Solvent blends.	complex mixture solvents 98% glycerine 0-2%		

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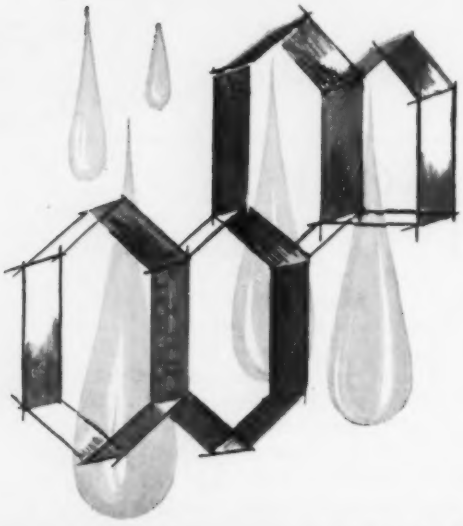

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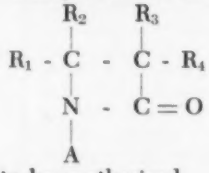
product that is left within the container, when the latter is nearly empty, without causing an alteration in the characteristics of the emergent stream.

Another valve which is finding increasing use is the one commonly referred to as a "metering valve" and this may be found both for space and surface sprays, employing all types of propellants, or for stream dispensing, when employing compressed gases. As with the standard valves, it is sometimes necessary to resort to breakup spray actuators in order to improve the spray characteristics, and very often special applicators are attached to the actuators in order to achieve the best product distribution. Special applicators are, of course, also used for standard valves, but not to the same extent.

About the preparation and technical properties of some condensation products of fatty acids with amino-carboxylic acids.

Dr. W. Frank, Frankfurt-Hochst

It has been found that condensation products of fatty acids with β-lactames of the general formula



are easily to be synthesized.

$R_1 - R_4 - = C_1 - C_5$ - alkyl group which may be unsaturated and/or substituted or not, or aryl groups, or cyclo-alkyl groups

A = H or alkyl group of max. 4 C-atoms.

They are prepared by reacting fatty acids with appropriate β-lactames under ordinary pressure and at temperatures between 50° and 200° Celsius. Their technical properties are similar to those of the sarcosine or polypeptide derivatives of fatty acids. These compounds exhibit good foaming and laundering properties and hard water stability.

The testing of cosmetic products on animals.

Dr. O. Jacobi, Wiesbaden

At first are the possibilities outlined of animal tests for efficacy as well as for irritation studies.

The fact of fundamental differences between the human skin and that of animals is discussed. There are anatomical differences and the sensitivity toward toxic material is also not the same.

Several examples demonstrate the cases in which animal experiments already have been used to test the efficacy of cosmetic products.

Methods used to check dermal toxicity are named and described. The evaluation of the reactions resulting from the tests on animals on the basis of scores is shown in tables. The text of the speech is illustrated by several pictures.

Animal tests cannot replace tests on the human skin. Even though there is no complete correlation between skin tests on men and animals the use of animal serves

a very important function. It serves the purpose to screen new materials and new formulars as well as the different steps while a new formula is developed.

If a substance or cosmetic product shows severe toxic or irritating properties men should not be exposed to it. In the opposite case if an animal test is negative it cannot be concluded that the material will be innocuous on the human skin too. A test on human skin should be performed in such a case.

Contribution to the study of the experimental depigmentation.

Dr. Robert Brun, Genf.

Experiments conducted on colored guinea-pigs have shown that the depigmentation induced by the monobenzyl-ether of hydroquinone was not a property of this substance alone. Another derivative of hydroquinone, the monomethyl-ether, also possesses this property but to an even greater extent. Other diphenols, both free or substituted, as well as currently used antioxidants were shown to have no depigmenting action. A few of these substances did reveal some activity, the accompanying irritation made the exact nature of such very difficult to evaluate.

The in vivo action mechanism of hydroquinone mono-ethers was investigated.

The depigmentation process seems to develop as follows: The basal layer of the epidermis is the first to be depigmented, with the superficial layers following last. Even when the depigmentation of the epidermis is complete, the melanophores (chromatophores) are still present in the dermis.

Should the treatment be stopped once the depigmentation is complete, one observes the rapid reappearance of new melanine granules in the days that follow. Whereas should the treatment be prolonged to twice the former time, this reappearance of the melanine occurs only after weeks or months.

With a short treatment (5 days), the Dopa-reaction was positive even though the epidermal depigmentation was almost complete. However, with a prolonged treatment (30 days), the Dopa-reaction became negative.

Furthermore, by means of in vitro experiments with potatoes tyrosinase, we can show that hydroquinone mono-ethers can be used as substrates by this enzyme. Thus, in a first phase of the reaction they can compete with the natural substrates. The final product of the reaction tyrosine-tyrosinase in presence of hydroquinone mono-ether is a red substance which is soluble and stable. According to a chromatographic analyse, this colored product, which is considered by some authors to be the Raper's red substance and by others to be the oxidation product of the mono-ether itself, is probably a compound comprising both oxidized tyrosine (before closure of the indol ring) and oxidized mono-ether.

Assay of lyophylized human placenta extract.

Dr. W. Colburn, USA

An assay method, based on the alkaline phosphatase enzyme activity, is presented, together with a critical evaluation of the effects of all the variables involved. The influence on the assay results of various preservatives, solvents, antioxidants, cosmetic ingredients, and processing conditions is included in the paper.

Inactivation of preservatives by nonionic emulsifiers.

Johanna Poprzan, Detroit

It is commonly believed that inactivation of preservatives by nonionics is the result of a complex formation between the hydroxyl group of certain preservatives and the polyether groups of the surfactants. This paper deals with the role played by various polyols and aliphatic alcohols in the prevention of inactivation of the preservative by non-ionic surfactant solutions. The antimicrobial activity of the simple aliphatic alcohols is well known. Propylene glycol is used as a preservative in the food and pharmaceutical industries. Low concentrations of these materials were found to be effective in preventing microbial growth when used in conjunction with methyl paraben. This action appeared to be of an additive type when judged by bacteriological tests. Other tests to be reported involve dialysis and light absorption, thus further elucidating the action of the alcohols and polyols on the non-ionic preservative system.

The swelling of hair in cold permanent waving agents investigations in polarized light.

Dr. F. Keil, Berlin

For permanent waving, alkaline thioglycollate solutions are used or neutral or slightly acid agents. It is said that with the latter, the set will last longer in a damp atmosphere, and this is supposed to be due to reduced hygroscopicity.

Much has been written about the absorption of water by hair, diameter determinations, expansion and contraction measurements, but these do not give a picture of the actual process of swelling. This process can, however, be observed under the polarizing microscope, Hair behaves in polarized light as an uniaxial crystal, i. e., it shows, when observed normal to direction of growth, strong interference colours; the actual colours observed depend on the thickness and refractive indices. It is worth remarking that on swelling the polarization colours change to those of a lower order. With the increase of diameter, this can only be explained by the refractive indices approaching each other. The hair thus approaches isotropy more and more. The "tonofibrils", forming the fusiform cells of the outer edge, or the keratin molecules become disorientated. From the gradual changes in interference colours, the penetration of the liquids in the submicroscopic field becomes visible, i. e. recognizable.

In the experiments under consideration solutions of various mercapto compounds, which had been adjusted to pH values of 3.7 - 9.7 by means of ammonia were used as standards, as well as water and ammonia. The hair samples are fixed on the mount by means of a loop, a few millimeters in diameter.

It becomes evident that swelling is slower at low pH values and is very much dependent on porosity, defects, and on mechanical and chemical damage. At pH 9.4, swelling in cold permanent waving solution

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is similar to that in water and is not even. One finds areas of differing swelling power. The liquids penetrate more easily the inner arc of bent hair than the outer arc. Areas of quick colour change, point to cavities and more severe damage.

In contrast, cold permanent waving solutions, at pH higher than 9.4, penetrate quickly and evenly from all sides into the hair. As the diameter increases, two sharply defined zones, starting at both edges, diffuse forward into the core of the hair. During this process, it takes on a uniform pale yellow interference colour, of a low order. If further swelling is interrupted at this stage, by means of hydrogen peroxide and/or weak acid, colours of a higher order appear again in the core of the hair. The effect is even stronger with formaldehyde. In the dried hair one finds again the original interference colours. If swelling takes place for a longer period, no polarizing colours of a higher order appear in the hair on after-treatment.

Differential layering, pointing to an ortho-para-cortex structure was not observed in straight hair.

The course of swelling of hair in water, when observed under the polarizing microscope, shows the extent of damage in the hair.

A number of coloured photomicrographs will be shown and the various phenomena will be discussed.

Contributions to the knowledge of the physical and chemical behaviour of human hair.

Dr. H. Freytag, Darmstadt

From a cosmetical point of view it is just as important to have an extensive knowledge of the properties of human hair as it is in textile technology with regard to the properties of the wool fibre. It has been recognized, that the experience gained in wool chemistry might be advantageously applied to human hair which has occasionally been included in such investigations; however, this idea has not been followed consequently enough. While wool of many individuals of one species is available for investigation and application, each species of human hair comes from an individual. This statistical difference and individual origin of human hair must be realized if the results of wool chemistry are to be applied. The experiments made with human hair refer to hygroscopic properties, acid bonding power and possible application of chemical test methods for wool after various hair treatments. The characteristic results obtained demonstrate again—and more thoroughly than those obtained with other methods—the high degree of variability of human hair. The findings are shortly discussed in connection with the well-known facts of wool-chemistry and with a view to cosmetic requirements of fundamental character.

Measurements on the spectral reflectance of oxydative dyeings.

Dr. H. Wilmsmann, Darmstadt

An objective measurement of colors is of importance for color-research as well as for the control of dyestuffs and dyeing-preparations, or imitation of given shades. The measurement of oxydative hair-dyeing requires partly grey human hair as test-material, because the resulting color depends on the oxydation-

dyestuff-component on the one side and on the partly bleached melanine-component on the other side.

A short survey of the special expressions of color-measurement as color-impression, stimulus, X,Y,Z-values and color-chart of the CIE-System introduces into a method of the measurement of dye-impression and stimulus of oxydative dyeings, special hair-dyeings.

The reflectance-spectralphotometer ZEISS-ELREPHO allows the measurement of the color-stimulus via the directional reflectance at seven wavelengths as well as the numerical statement of the dye-impression via direct measurement of the tristimulus values X, Y and Z with three color filters.

Measurements on the spectral reflectance of oxydative dyeings on wool fabrics, buffalo hair and medium blonde hair render clear the relations already well known in the practice of hair-dyeing. The reflectance of oxydative dyings with p-toluylenediamine combinations of p-toluylenediamine with m-aminophenol, some commercial available hairdyeing-preparations in "natural"—and "mixtones" and bleached and lighter-dyed hair is demonstrated and discussed.

It is proved that the so-called KUBELKA-MUNK-function is valid for oxydative dyeings in lower concentrations, too, thus the estimation of the dye-concentration in dyed fibres via the directional reflectance being possible. Due to the complicated formula of commercial hair-dyeing-preparations the analytical value of the KUBELKA-MUNK-functions resp. reflectance measurements is limited.

The early history of cosmetics in Mexico: some data.

Prof. J. Erdos, Mexico

Short introduction on cosmetics in early times.

Medicine and cosmetics in Mexican mythology. The goddess Tzapotlatena invents a resin called "uxitl" against skin complaints. Xochipilli, the god of flowers, Xipe, Tlaltecuin, patron of sick children, Cihuocoatl (who corresponds to Eve, as the first woman to give birth) Centeotl, earth goddess and goddess of medicinal herbs.

Discovery of Mexico (Hernan Cortes); two documents containing medicinal and cosmetic data: 1. Dr. Francisco Hernandez (Montalban, Spain, 1517-1587), who arrives with the first scientific expedition to Mexico (1570-1577); his publication "the History of Plants of New-Spain", publication on Hernandez by Dr. German Somolinos d'Ardois of recent date (Mexico). 2. "General History of Things in New-Spain" by Fray Bernardino de Sahagun (b. Spain 1500, since 1529 in Mexico, died 1590).

Some details of Mexican geography and climate accounting for the extraordinarily rich flora. F. Hernandez actually describes 2879 plants. Outline of development since 1521 (conquest): population of Mexico; number of cosmetic and pharmeceutical firms; some data concerning the Ministries of Health and Education, nursing homes, hospitals, the University and Technical Colleges.

Short account of cosmetic manufacture, pharmaceuticals (principally of plant origin), of pre-columbian times.

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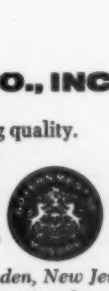

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PACKAGING & PROMOTION

1

1—Coty

"Little Miss Coty" is being introduced by Coty, Inc. for girls between the ages of 4 and 12. A prestige line of children's toiletries all items are packaged in deluxe, floral embossed packaging designed to appeal to the children's desire to be "grown-up". The popularly priced line is being offered in a counter unit with a 25 piece assortment of merchandise during the introduction period.

2

2—Simonetta

Incanto perfume and cologne are being introduced by Simonetta of Rome in an attractive gift package. A purse dispenser of perfume and a measured spray mist of cologne are packaged in golden metal containers topped with black caps. An oval box lined in velvet has reproductions of scenes of ancient Rome, done by the 18th Century artist, Piranesi, on the cover-wrap. The gift set will retail at $7.50.

3

3—Biotherm

Cosmetiques Biotherm of Fifth Avenue, New York is introducing the Biotherm line of skin cremes, formulated in France, in the United States. Packed in an aluminum tube with a white phenolic cap with a gold print collar the product is enclosed in set-up boxes with a raised partition which cushions the neck area of the tube. The cap is only partially threaded before purchase and on completely closing an internal fitment pierces a hermetic seal in the tube orifice, guaranteeing complete product freshness. The tubes are printed in pink and gold on a polished white background.

4—Helene Curtis

A dry skin bath oil has been introduced by Helene Curtis called "Tender Touch". Test marketed in Illinois, Wisconsin, Minnesota, Iowa and Indiana, the new product will be promoted as a light powder base and nightly facial lubricant as well as a bath oil. Television commercials beginning during the first part of October will emphasize the theme "Look Younger, Feel Smoother . . All Over".

4

5

6

5—Max Factor

Shadow Play is the name of a new container for Max Factor's eye shadow. A wand-type container holds four different Hi-Fi eye shadow shades in miniature sizes. Each shade is in an individual holder which fits snugly into the others to make the Eye Shadow Wand. When separated from the others, each shade is applied directly from its own holder. Shadow Play is being marketed in a two wand carton containing a total of eight different Hi-Fi colors.

6—Kings Men

A men's hairdressing has been introduced by Kings Men in a non-collapsible plastic tube. Packaged in a carton with a plastic window the tube is of arresting fire engine red. Promoted for all-day hair care, the hairdressing is claimed to control dandruff. The product will retail at $1.00 per tube.

7

7—Shulton

A new line of grooming aids for men will be introduced in October by Shulton, Inc. Called York Town 1781, introduction of the line will feature individual packages of Cologne for Men, and After Shave Lotion and Shaving Talc as part of a gift set. Packaging will be based on the early American theme. Bottle caps will be reproductions of George Washington's gold inaugural buttons. Bottles are replicas of early blue and green glass apothecary jars, embossed with tiny gold gun carriage.

8—Bourjois

Bourjois is now marketing Evening in Paris hand cream. Silicone fortified the cream is being promoted to smooth roughened skin and to soothe chapped hands. The urn-shaped oval jar has a label printed in blue and silver. An introductory offer of the $2.00 jar at $1.00 is being made. Extensive advertising will begin in October for the new hand cream.

8

RICHFORD offers the new shape...

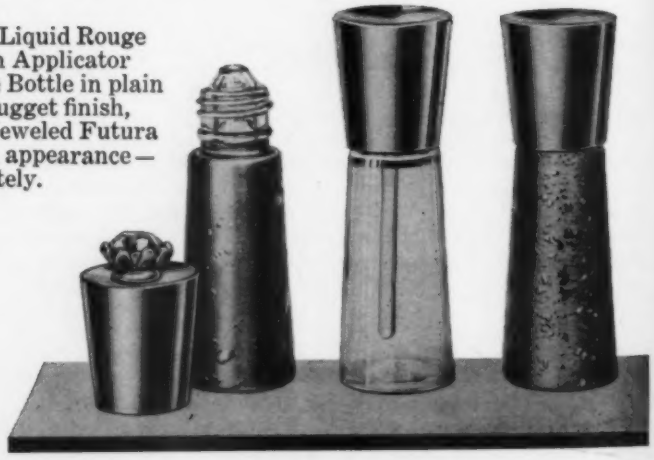
in Sachet Containers

A new flat oval 1½ oz, Jar, #1908—with or without Sifter Top and Goldcote Closure for sachet powder. And a companion 1/2 oz.,

#R-33, Liquid sachet bottle with Touch-N-Flo dispenser unit—gold metal protector cap. (Patented)

For Cream Sachets in 1/2 oz. Exquisite Bottle with gleaming Goldcote Futura Cap. In clear glass or its new Gold Nugget or Gold Mist (Brocade) finish. Cap has Applicator Rod as shown.

A new 2 dram Liquid Rouge Container with Applicator Rod. Exquisite Bottle in plain or new Gold Nugget finish, with plain or Jeweled Futura Cap. Deluxe in appearance—priced moderately.



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. . NEWS AND EVENTS . . . NEWS AND

Color Additive Amendments Approved — Delaney Cancer Clause Included

Congress has approved the Color Additive Amendments to the Federal Food, Drug, and Cosmetic Act which authorizes the use of suitable color additives in or on foods, drugs, and cosmetics in accordance with regulations to be issued by the Secretary of Health, Education, and Welfare, prescribing the conditions, including maximum tolerances, under which such color additives may be safely used. The amendments, as they go to the White House, include the controversial Delaney anticancer clause which provides that a color additive shall be deemed unsafe and shall not be listed for any use which will or may result in ingestion of all or any part of such additive if the additive is found to induce cancer when ingested by man or animal, or if it is found to induce cancer in man or animal by other tests, not involving ingestion, which are considered to be appropriate for the evaluation of the safety of additives for use in food.

The amendments are designed to meet a pressing need for replacing the inconsistent, outmoded provisions governing the use of different kinds of certified colors for foods, drugs, and cosmetics with a scientifically sound and uniform system of listing of color additives of any kind which may safely be used in foods, drugs, or cosmetics, subject, when necessary, to appropriate tolerance limitations and other conditions of use and to official certification of batches of colors so as to assure the safety of such use to the consumer.

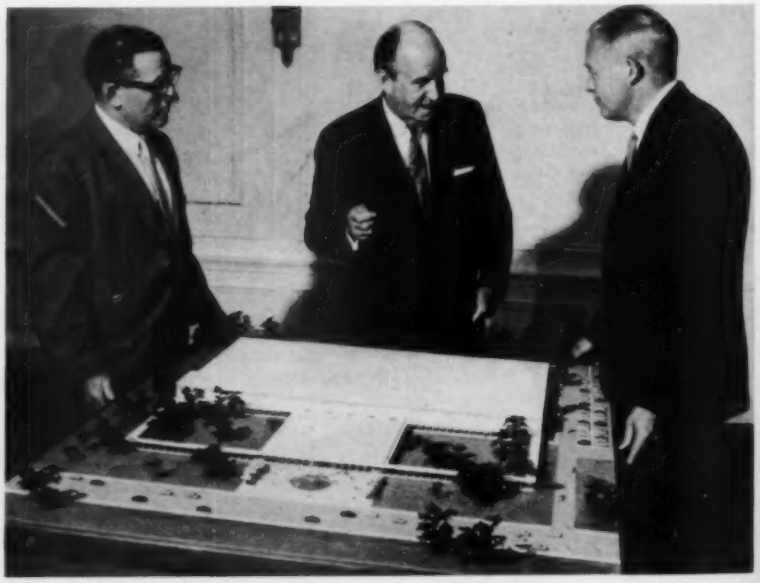
Symposium on science in advertising set for October

The Association of Consulting Chemists and Chemical Engineers will sponsor a symposium on October 25th at the Hotel Shelburne, New York City, on "The Role of Science in Advertising." Panelists include: Earl W. Kintner, chairman, Federal Trade Commission, Kenneth B. Willson, president, National Better Business Bureau and S. R. Bernstein, editorial director, *Advertising Age*.

The symposium, which begins at 5:30 PM, will be a discussion on how laboratory tests and other scientific counsel are used in stressing public, scientific and advertising interest and will discuss the effect of regulatory efforts.

DCAT holds 70th annual meeting at Lake George

The Drug, Chemical and Allied Trades Association (a section of the New York Board of Trade, Inc.) held its 70th annual meeting on September 15-17 at the Hotel Sagamore, Bolton Landing, Lake George, New York. Principal speakers at the September 16th business session were, Dr. Morris A. Jacobs, commissioner of hospitals of the City of New York, and Dr. William B. Walsh, president of project HOPE. On Thursday, September 15th an open discussion followed a presentation by Warren Freedman, counsel for Clairol, Inc., on "The 1960 Cutter Decision—A Lesson in Judicial Lawmaking: No Fault = Liability."



Edward Durell Stone, center, world-famous American architect, discusses the model of the new Yardley plant which he designed with Joseph A. Rosenthal, left, president of Engineers, Inc., the Newark firm which provided the engineering design for the plant which will go up in Totowa, New Jersey, and Louis Brennesholtz, vice president in charge of production of Yardley of London, Inc. The three million dollar structure will house finishing and distributing facilities for Yardley products sold in the United States. A 50 percent increase over present Yardley facilities will result from completion of the 236,000 square foot building.

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3Ms and Warner-Lambert negotiate for merger

Minnesota Mining & Manufacturing Company and Warner-Lambert Pharmaceutical Company have announced that they are engaged in negotiations for the purpose of combining the two companies. Approval of the two companies' boards of directors and stockholders will be needed before the combination can be achieved.

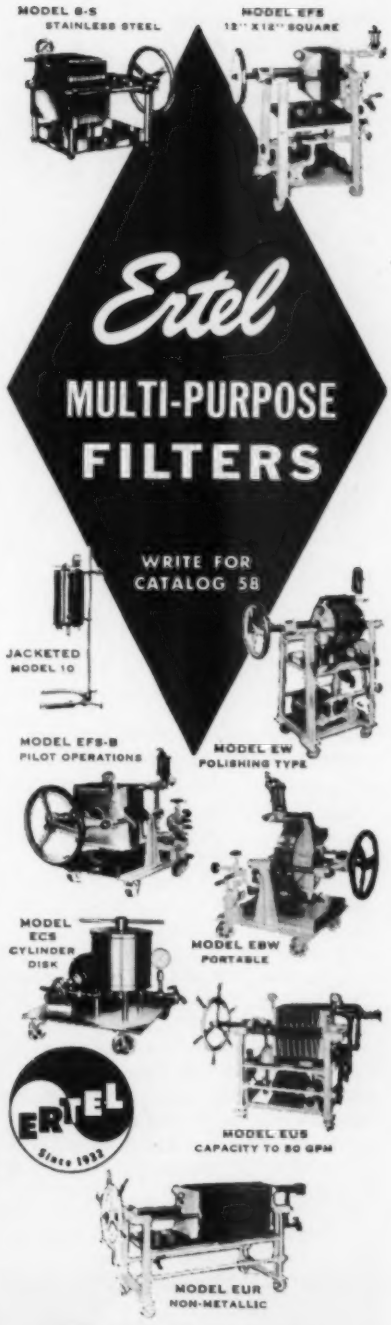
Minnesota Mining & Manufacturing Company has been engaged in extensive research some of which is expected to lead to development of chemicals and other products which may find future use in medicine and pharmacy. Emphasis at Warner-Lambert is on research and marketing of ethical and proprietary pharmaceuticals although the company's product line is diversified.

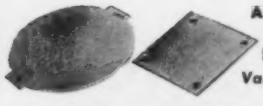
Paris party for CCS members enroute to Munich, Germany

Gattefosse S.F.P.A. of Lyon, France and R.I.T.A. Chemical Corp., Chicago, Illinois were hosts to members of the Cosmetic Chemists Society at an informal champagne party on August 24 at the Claridge Hotel in Paris, France. The cosmetic chemists, now touring Europe, are enroute to the International Seminar at Munich, Germany. This was the first stopover on a tour which will take them to several European Capitals.

(Left to right) S. T. Goode, R.I.T.A. Chemical Corp., L. Conrad, American Cholesterol Products, and M. Sliedrecht, Von Schuppen and Zoon, Holland.

S. T. Goode, R.I.T.A. Chemical Corp., M. Sliedrecht and Dr. P. Mulder, Von Schuppen & Zoon, Holland and M. G. deNavarre, Cosmetic Laboratories, and editorial director of American Perfumer are shown from left to right.





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Denver drug company bought by Kansas City firm

Lee Drug Company, Denver, Colorado, has been purchased by Lucky Tiger Manufacturing Company, Kansas City, Missouri. In July Lucky Tiger announced the acquisition of the York Pharmacal Company of St. Louis, Missouri. Lucky Tiger now has service facilities for its wet and dry drug line in key cities to cover the entire midwest.

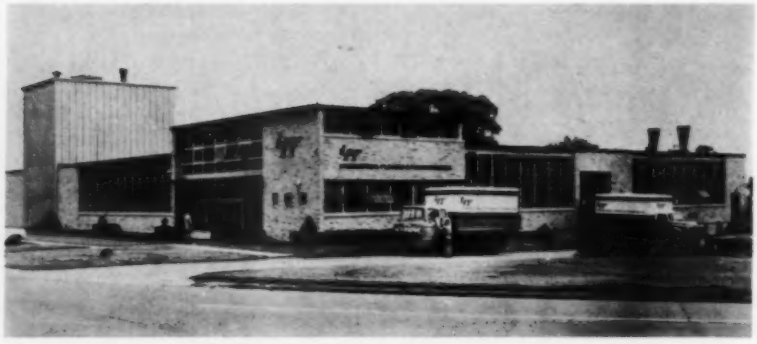
Fleuroma offers overseas service

Fleuroma, Inc. now offers an integrated international operation to American customers who want special services abroad. The following facilities are available to those who want such service: Poons and Lengsfelder, Ltd., 12 Broadway, London, S.W. 1, England, manager John King; Fleuroma, S.A.R.L., 18 Rue Marbeuf, Paris 8, France, manager Lucien Romagnan; and Essencias Fleuroma Limitada, 5107 Av. Washington Luiz, Sao Paulo, Brazil, manager Leo Schulmann.

Kohnstamm sets up manufacturing agreement with English firm

Louis J. Woolf, chairman of the board of H. Kohnstamm & Company has announced a joint agreement with Horace Cory and Co., Ltd. of London whereby Cory will manufacture Kohnstamm dyestuffs and pigment colors for sale in the Commonwealth and other specified countries of the world. The London manufacturer is one of the largest pigment producers in Great Britain.

A new plant will be constructed on the outskirts of London and will be equipped with modern production facilities for the manufacture of the products under the Kohnstamm label and under Kohnstamm's formulations and supervision.



This flavor plant at Teterboro, New Jersey will be expanded on a three year program to lead to the consolidation of all of International Flavors and Fragrances U.S. flavor activities at this location. Over 14,000 square feet of production space will be added at the Teterboro plant during the next six months. An administration building for the flavor division and a research center will be added. Both of these are now headquartered at Elizabeth, New Jersey.

Naarden establishes subsidiary company in Mexico

N. V. Chemische Fabriek "Naarden" has announced that a subsidiary company has been established in Mexico under the name Naarden de Mexico, S.A. Production of flavors and perfume-compounds for Mexican industry will be supplemented by manufacture of food preservatives benzoic acid and sodium benzoate in cooperation with Salicilatos de Mexico, S.A.

Glycerine awards to include product application

The Ninth Annual Glycerine Research Awards will give special recognition and emphasis to practical applications for glycerine and its derivatives. Two awards of $750 and an honor plaque will be given in January, 1961 for outstanding work in basic research and for practical glycerine application. Established in 1952 by the Glycerine Producers' Association, the awards are given annually for new and independent research contributing to knowledge and use of glycerine and its derivatives.

Canadian Chemical Specialties Manufacturers to convene

The third annual meeting and conference of the Canadian Manufacturers of Chemical Specialties will be held at the Queen Elizabeth Hotel, Montreal, October 24 through 26.

Meetings on Monday will consist of planning sessions for programs for the coming year. A panel on tariffs will take place Tuesday morning. Tuesday afternoon will feature a panel on pesticides, and forums on waxes and floor finishes, aerosols and automotives, and on soaps and detergents and disinfectants and sanitizers.

The final day, Wednesday will have a panel on marketing during the morning, and forums on the subjects covered during the Tuesday afternoon sessions.

Further information on this convention may be obtained from the association at 3405 Cote Des Neiges Road, Montreal 25, Canada.

PERSONALITIES

Robert S. Lubin

Robert S. Lubin has been elected president of Lesli Laboratories, Inc. Mr. Lubin comes to Lesli from American Sound Products, Inc. where he was president of this firm, manufacturers of electronic hearing aids.

Mr. Lubin has been instrumental in founding Lesli Laboratories specifically for the purpose of marketing a professional home facial kit. He plans to introduce this kit on a market-by-market penetration supported by newspaper and television advertising together with cooperative advertising.

Henry Jones has been appointed field sales manager in charge of chain drug store operations for Helena Rubinstein, Inc. Mr. Jones was most recently with Sylvania Electric Products, Inc., as sales manager of the Tube Division, but prior to that had spent many years in the cosmetics industry.

Leo J. McNamara has been appointed assistant to the president of the Lady Esther division of Chemway Corporation. Mr. McNamara, who has been with Chemway since 1952, will expand Lady Esther's marketing program in his initial assignment in the area of sales administration.

E. T. Bush, Jr. has joined the staff of Albert Verley & Company, processors of essential oils and aromatic chemicals.

Mr. Jean Warner Baer is now manager of manufacturing for Julius Kupfer, Inc., Elmhurst, L. I., New York. Mr. Baer joins Kupfer from Sprapak Chemicals where he was manager. In his position at Kupfer he will be responsible for production and control of all present operations and the development of further expansions.

Herbert D. Rosenthal has been named vice president-sales for Dorothy Lamour, Inc. Mr. Rosenthal has been in the cosmetic industry for 26 years serving as president of Patricia Stevens Cosmetics, general sales manager for Helena Rubinstein and executive vice president for both Chen Yu and Pinaud, Inc.

Edward Stern

Edward Stern will fill the newly created position of director of marketing at Crown Cork & Seal Company. Mr. Stern will responsible for evaluation and marketing of all present and new products, marketing techniques, pricing, sales training, advertising and promotion.

Harold J. Rosen has been elected vice president of Helene Curtis Industries, Inc. Mr. Rosen, who has been with the company since 1944, was formerly executive assistant to the board chairman. In his new position Rosen will be responsible for corporate acquisitions and development activities.

Charles L. Lightfoot has been appointed vice president, marketing for Goldschmidt Chemical Corporation. In his new position, Mr. Lightfoot will be responsible for sales of existing products and development and marketing of new products.

Lowell A. Ledgett will succeed the retiring Earl J. Reddert as director of engineering for the Colgate-Palmolive Company. Mr. Ledgett joined Colgate in 1934 and has been manager of engineering and construction since 1958.

Dr. James Fisher and Dr. Frank Rupp, Jr. have been appointed to the Biological Research Laboratories of Colgate at New Brunswick, New Jersey.

Dr. Sam Yankelowitz will join the staff of Colgate-Palmolive's Oral Health Research Laboratories. Janet Massing has also been appointed to the staff of the Oral Health Research Laboratories of Colgate.

Robert W. Montgomery has joined Verona-Pharma Chemical Corporation as sales manager of the Verona Aromatics Division. Mr. Montgomery was formerly sales manager of Fritzsche Brothers, Inc.

B. Wellenkamp, former sales manager of the Verona Aromatics Division, has been granted partial retirement at his request. Mr. Wellenkamp will, however, continue to participate in Verona's activities.

Robert W. Montgomery

Richard A. Dey has been elected secretary of Shulton, Inc. by that company's board of directors. Mr. Dey joined Shulton in 1944 as personnel manager and has served as director of industrial relations since 1955.

Nicholas F. Scodari has joined The Mennen Company as senior research chemist. Mr. Scodari was formerly associated with Block Drug Company of Jersey City, N. J. and Carroll, Dunham, Smith of New Brunswick, N. J.

Mrs. T. M. Sayman, president of the Sayman Products Company of St. Louis, was presented with an Honorary Award as Commander of the British Empire (C.B.E.). The award, presented by His Excellency The British Ambassador, is in recognition of her promotion of friendly British-American relations over a period of many years.

Peter Smithers, a member of the British Parliament, and Mrs. Sayman's son-in-law, has been appointed a member of the British Delegation to the General Assembly of the United Nations.

John C. Fedoruk has been named technical director of the General Chemical Division of Allied Chemical Corporation. Mr. Fedoruk succeeds Dr. Howard H. Hurmence who was recently advanced to assistant director of research and development for the Corporation.

Obituary

Ross LeB. Daggett, vice president, George Lueders & Company, died of a heart attack on July 31st.

John J. Reidy

John J. Reidy will fill the newly created position of vice president in charge of marketing at Yardley of London, Inc. Mr. Reidy will coordinate the functions of the sales, merchandising and advertising departments of the company. He comes to Yardley from Schick, Inc. where he has been an officer of the company since 1958.

Gerbert G. Reid has been named controller of International Flavors and Fragrances, Inc. He was previously associated with Price Waterhouse & Company.

Walter P. Kuenstler, director of marketing development Warner-Lambert Pharmaceutical Company's consumer product divisions has been appointed to the new post of executive vice president, Hudnut-DuBarry division. George J. Abrams has resigned as president of the Hudnut-DuBarry division. His future plans will be announced later.

Wayne E. Edwards has been elected vice president, manufacturing and research of The Allen B. Wrisley Company. Mr. Edwards was formerly director of research for the company.

Mr. Francois Camilli

Obituary

Mr. Francois Camilli, President and General Director of Camilli, Albert & Laloue S.A. died on July 16th at his home in Grasse after a long illness at the age of 66. He was a Chevalier of the Legion of Honour, President of "Les Cultures Florales Mediterraneennes", Charter-Member of the "Comite Consultatif du Groupement Interprofessionnel des Fleurs et Plantes Aromatiques" (G.I.F.P.A.) and Honorary President of the 'Amicale des Corses de Grasse". Mr. Camilli has visited the United States on several occasions and in 1950 established the American Subsidiary Camilli, Albert & Laloue, Inc. Mr. Camilli is survived by his widow Mrs. Camilli and his son Dominic. Mrs. Camilli has joined the Board of the Grasse concern as a Director and Mr. R. M. Smith, a Director for 22 years was appointed President and Managing Director.

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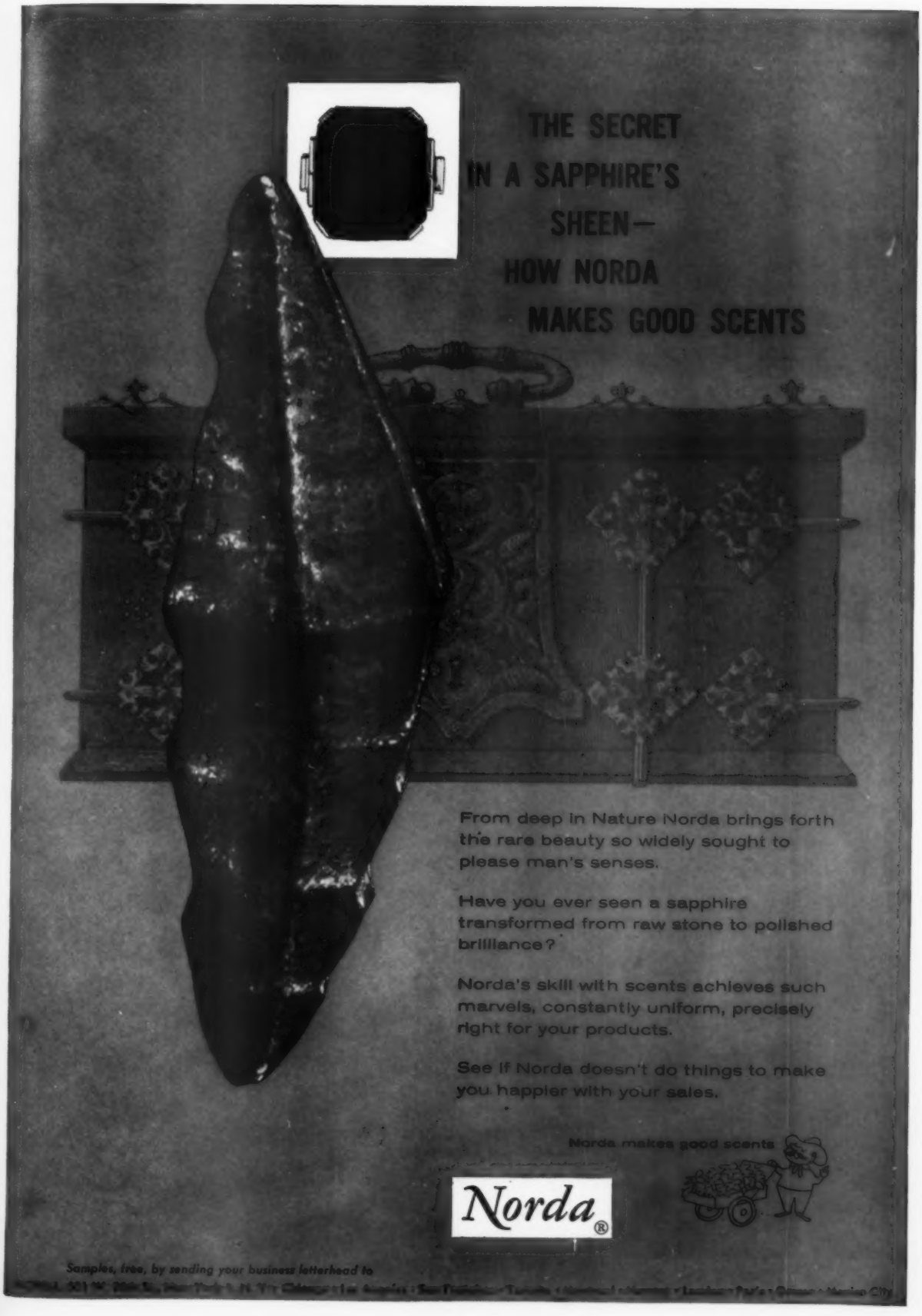
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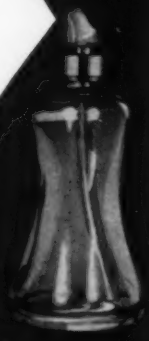

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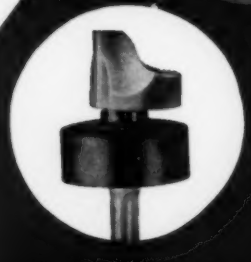

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